# Mixed Metal Oxide Nanomaterials: Synthesis Strategies, Structural Properties and Catalytic Applications- A Review

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**Abstract**

Mixed metal oxide (MMO) nanomaterials have attracted extensive research interest over the past decade due to their exceptional catalytic, electronic, and structural properties. Incorporation of two or more metal cations into a single oxide lattice enhances redox behavior, oxygen mobility, and structural stability, distinguishing MMOs from single-metal oxides. These characteristics enable their widespread application in heterogeneous catalysis, environmental remediation, energy conversion, and industrial chemical reactions. This review provides a detailed analysis of synthesis strategies, structural properties, and catalytic applications of MMOs, focusing on works published. The interrelationship between synthesis methods, structural features, and catalytic performance is critically examined, and key challenges and future research directions are discussed.

**Keywords:** Mixed metal oxides, Nanomaterials, Catalysis, Structural properties, Synthesis methods

## 1. Introduction

Nanostructured metal oxides have revolutionized heterogeneous catalysis by providing a high surface area, tunable electronic structure, and unique surface chemistry. Among these, mixed metal oxide (MMO) nanomaterials, composed of two or more metal cations in a single lattice, exhibit synergistic effects that lead to superior catalytic performance, thermal stability, and redox flexibility compared to single-metal oxides (Busca, 2014). The combination of metal ions allows modulation of lattice parameters, electronic structure, and oxygen vacancies, which are critical in catalytic reactions such as oxidation, reduction, and reforming processes. MMOs also exhibit varied crystal structures, including spinel, perovskite, and layered double oxides, each offering specific advantages in terms of surface reactivity and stability.

Environmental concerns, energy conversion challenges, and industrial process optimization have driven interest in MMO nanomaterials. They have been used for catalytic degradation of volatile organic compounds (VOCs), oxidation of CO, water splitting, and fuel cell reactions. Understanding the correlation between synthesis methodology, structural features, and catalytic performance is vital to rational design of efficient MMO nanocatalysts. While numerous reviews have focused on single-metal oxides or perovskites, this review aims to comprehensively summarize MMOs across multiple synthesis techniques, structures, and applications published between 2010 and 2016.

**2. Objectives of the Review**

The main objectives of this review are:

1. To analyze synthesis strategies employed for MMO nanomaterials.
2. To examine structural and physicochemical properties influencing catalytic activity.
3. To summarize major catalytic applications in environmental, energy, and industrial domains.
4. To correlate synthesis methods with structure–property–performance relationships.
5. To identify challenges in the current research and outline potential future directions.

**3. Methodology of Literature Review**

This review is based on a systematic analysis of peer-reviewed publications that report experimental synthesis, characterization, and catalytic performance of MMOs. Relevant journals included *Applied Catalysis A: General*, *Catalysis Today*, *Journal of Physical Chemistry C*, *ACS Catalysis*, and *Chemical Reviews*. Selection criteria focused on studies providing detailed structural characterization (XRD, SEM, TEM, BET, XPS), well-defined synthesis protocols, and catalytic evaluation under controlled conditions. Comparative analysis was performed to identify trends in synthesis–structure–activity relationships and recurring challenges across the literature.

**4. Synthesis Strategies of Mixed Metal Oxide Nanomaterials**

**4.1 Co-precipitation Method**

Co-precipitation remains one of the most widely used methods for MMO synthesis due to its simplicity, scalability, and ability to produce homogeneously mixed cations. In this method, aqueous solutions of metal salts are simultaneously precipitated by adding a base under controlled pH and temperature conditions, followed by filtration, drying, and calcination. The method enables atomic-level mixing of metal ions, which enhances metal–metal interactions and redox performance (Li, Li, & Zhang, 2012). A significant advantage of co-precipitation is the ability to tune particle size and composition by adjusting precursor concentrations and precipitation conditions. However, challenges such as agglomeration, irregular morphology, and difficulty in controlling porosity often arise. Several studies have addressed these limitations by using surfactants or chelating agents during precipitation to control particle growth and prevent aggregation.

For example, Ni–Co oxide synthesized via co-precipitation exhibited a spinel structure with uniform particle size distribution and high surface area, which translated into excellent oxygen evolution reaction (OER) performance (Roy, Hegde, & Madras, 2011). Similarly, Cu–Mn mixed oxides prepared using co-precipitation demonstrated high activity for VOC oxidation due to the homogenous distribution of Cu and Mn ions and the resulting defect-rich lattice, which promoted oxygen mobility (Santos, Pereira, & Órfão, 2014).

**4.2 Sol–Gel Method**

The sol–gel technique provides precise control over stoichiometry, homogeneity, and particle size

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distribution. Metal precursors, usually metal alkoxides or salts, undergo hydrolysis and condensation to form a three-dimensional gel network. Upon calcination, this gel transforms into nanostructured MMO materials with high surface area and controlled porosity (Santos et al., 2014). The sol–gel process allows tailoring of the particle morphology and pore structure, which is essential for catalytic applications that rely on accessible active sites.

Studies have demonstrated that Fe–Ce oxides synthesized by the sol–gel route exhibit well-dispersed nanorods with abundant oxygen vacancies, which enhanced CO oxidation activity (Zhao, Li, & Wang, 2013). The sol–gel method also facilitates the incorporation of dopants to fine-tune surface acidity or basicity, providing an additional handle to optimize catalytic behavior for specific reactions such as selective oxidation and hydrogenation.

### 4.3 Hydrothermal and Solvothermal Methods

Hydrothermal synthesis involves the crystallization of MMOs in aqueous media under high pressure and temperature in sealed autoclaves. This method yields highly crystalline nanostructures with controlled morphology, including nanorods, nanowires, nanosheets, and hierarchical assemblies (Zhao et al., 2013). The hydrothermal approach enables precise control over particle size, crystallinity, and phase purity, which are crucial parameters for catalytic efficiency. Similarly, solvothermal synthesis using organic solvents can further influence particle morphology and surface chemistry, providing nanomaterials with tailored active sites for catalysis.

For instance, hydrothermally synthesized Fe–Ce oxide nanorods exhibited high surface oxygen vacancy concentration and strong reducibility, leading to enhanced catalytic performance in CO oxidation and VOC degradation (Roy et al., 2011). Hierarchical structures prepared via solvothermal methods also showed improved mass transport and higher surface area, which are beneficial for heterogeneous catalysis.

### 4.4 Combustion and Microwave-Assisted Methods

Combustion synthesis is a rapid, energy-efficient technique that employs exothermic reactions between metal nitrates and organic fuels. This method produces highly porous MMO nanomaterials with high defect density and enhanced redox activity (Varma et al., 2011). The high defect concentration facilitates oxygen mobility and surface reactivity, improving catalytic performance in oxidation reactions.

Microwave-assisted synthesis is another rapid method that provides uniform heating and accelerates nucleation and crystallization. Microwave-assisted MMOs often exhibit smaller particle sizes and narrower size distributions compared to conventional thermal methods. The combination of microwave-assisted heating and combustion has been shown to produce Ni–Co oxides with superior OER activity due to enhanced surface area, defect density, and improved charge transfer characteristics (Suntivich et al., 2011).

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## 5. Structural and Physicochemical Properties

The catalytic efficiency of MMO nanomaterials is strongly governed by structural and physicochemical properties, including crystal structure, particle size, surface area, porosity, and electronic configuration. X-ray diffraction (XRD) provides information on phase composition and crystallinity, while electron microscopy (SEM, TEM) reveals particle morphology, size, and aggregation state. Nitrogen adsorption-desorption analysis (BET) assesses surface area and porosity, which directly impact catalytic accessibility.

Incorporation of multiple metal cations induces lattice distortion, oxygen vacancies, and defect sites, which are critical for redox reactions. These structural defects serve as active sites for adsorption and activation of reactants (Busca, 2014). Surface acidity and basicity, measured via temperature-programmed desorption of $NH_3$ or $CO_2$, further influence selectivity and reaction pathways. For example, Cu–Mn mixed oxides with defect-rich lattices exhibited enhanced oxygen mobility and higher catalytic activity for VOC oxidation (Santos et al., 2014). Similarly, Fe–Ce oxides with abundant oxygen vacancies demonstrated excellent performance in CO oxidation due to facile lattice oxygen participation.

## 6. Catalytic Applications of Mixed Metal Oxide Nanomaterials

### 6.1 Environmental Catalysis

MMOs have been extensively used in environmental applications, including oxidation of CO, degradation of VOCs, and removal of nitrogen oxides. Their high oxygen mobility, redox flexibility, and defect-rich surfaces enable efficient pollutant conversion under mild reaction conditions (Roy et al., 2011). For instance, Ni–Co oxide spinels have been applied for catalytic oxidation of CO and VOCs, demonstrating superior activity due to cooperative interactions between Ni and Co ions and the resulting lattice oxygen availability.

Fe–Ce oxides, with nanorod morphology, have shown excellent performance in CO oxidation and VOC degradation, attributable to the synergistic effects of Fe and Ce ions and the high concentration of oxygen vacancies. Layered Zn–Al oxides have also been utilized for VOC degradation and biodiesel production, demonstrating the importance of layered structures and high surface area in environmental catalysis (Santos et al., 2014).

### 6.2 Energy Conversion and Storage

In energy-related applications, MMO nanomaterials serve as electrocatalysts for fuel cells, water splitting, and biomass conversion. The tunable electronic structure and high defect density of MMOs enable efficient charge transfer and stable catalytic behavior under operating conditions. Perovskite-type MMOs, such as La–Sr–Mn oxide, have been widely investigated as cathode materials in solid oxide fuel cells, demonstrating high oxygen reduction reaction activity and long-term stability (Suntivich et al., 2011).

Hydrothermally synthesized Ni–Co oxides and mixed spinels have also been explored as

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electrocatalysts for oxygen evolution reaction (OER), showing improved current density and lower overpotential compared to single-metal oxides. These improvements are attributed to enhanced active site density, better electron conductivity, and favorable surface morphology.

### 6.3 Industrial Chemical Catalysis

MMOs are widely employed in selective oxidation, hydrogenation, and dehydrogenation reactions. The combination of different metal ions allows tuning of surface acidity and redox potential, leading to improved selectivity and suppression of undesired side reactions (Wachs, 2011). For example, Cu–Mn and Ni–Co oxides have been applied for selective oxidation of hydrocarbons, while Zn–Al and Ti–V oxides are employed in biodiesel production and photocatalytic reactions.

The superior thermal stability of MMOs enables their use under harsh industrial conditions, where single-metal oxides often undergo sintering or deactivation. Furthermore, the hierarchical structures and high porosity achieved via sol–gel or hydrothermal synthesis enhance mass transport and accessibility of active sites, improving catalytic efficiency in industrial reactions.

### 7. Summary Table

**Table 1. Synthesis methods, structural features, and catalytic applications of mixed metal oxide nanomaterials**

| MMO System | Synthesis Method | Structural Characteristics | Catalytic Application |
|---|---|---|---|
| Ni–Co Oxide | Co-precipitation | Spinel structure, high surface area | Oxygen evolution reaction |
| Cu–Mn Oxide | Sol–gel | Defect-rich lattice, nanoscale particles | VOC oxidation |
| Fe–Ce Oxide | Hydrothermal | Nanorods, oxygen vacancies | CO oxidation |
| Zn–Al Oxide | Combustion synthesis | Layered oxide, porous morphology | Biodiesel production |
| La–Sr–Mn Oxide | Sol–gel | Perovskite structure, high crystallinity | Fuel cell cathode |

### 8. Challenges and Future Perspectives

Despite significant progress, MMO nanomaterials face challenges in precise control over composition, phase purity, and morphology, especially during scale-up synthesis. Particle aggregation, inconsistent defect density, and lack of reproducibility hinder their widespread industrial adoption. Future research should focus on combining advanced synthesis techniques with computational modeling to predict structure–activity relationships. Operando characterization methods, such as XPS, in-situ Raman, and EXAFS, are essential to monitor active sites during catalysis and understand dynamic

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surface phenomena.

Integration of machine learning and data-driven approaches for rational design of MMOs could accelerate discovery of new high-performance catalysts. Additionally, developing eco-friendly and low-energy synthesis methods is vital for sustainable production of MMO nanomaterials.

**9. Conclusion**

Mixed metal oxide nanomaterials are a versatile and promising class of catalysts with applications spanning environmental remediation, energy conversion, and industrial chemical processes. Their superior catalytic performance arises from synergistic metal–metal interactions, defect-rich structures, and tunable physicochemical properties. Synthesis methods such as co-precipitation, sol–gel, hydrothermal, and combustion approaches play a critical role in determining particle morphology, defect density, and surface reactivity. By establishing clear correlations between synthesis, structure, and catalytic performance, researchers can rationally design MMO catalysts with enhanced efficiency and stability. Continued advancements in synthesis, characterization, and computational modeling will be key to unlocking the full potential of MMO nanomaterials.

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