A SURVEY: SYNTHESIS OF MAGNETIC OXIDE SYSTEMS

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Abstract

Nanocrystalline magnetic materials have attracted considerable interest due to their uniqueness and remarkable properties in various fields including physics, chemistry, biology, medicines,material sciences and engineering. Global scientific and research groups are interested in magnetic nanoferrites among magnetic oxides because of their unique characteristics, which are very helpful for designing and developing magnetic devices, sensors and actuators, magneto-electronics, electrochemical and electromechanical systems. There are different methods of synthesis of magnetic oxide systems like dry method and chemical method. Some of the few chemical methods are Co-precipition, Sol-gel, thermal decomposition and combustion etc.The focus of this article is comparative study of the synthesis of magnetic oxide systems.

Keywords- Nanoferrites, Combustion, Co-precipitation,Sol-gel

1. Introduction

Metal Oxides nanoparticles (MONPs)exhibit unique physical,chemical,optical and electronic properties .Compared to their bulk counterparts, nanomaterials have particle size in the 1–100 nm range and a high surface to volume ratio that establishes various or improved thermal, mechanical, optical, electrical, magnetic, and reactive qualities [1]. While in the case of bulk materials, the chemical composition is the main factor that determines their properties, in the case of nanomaterials, besides the chemical composition, the particle size and morphology determine most of their characteristics. Moreover, these properties can be tuned based on the particle size and chemical composition [1,2].

Among the magnetic oxides; magnetic nanoferrites attract the attention of global scientific and research communities for their fascinating properties and phenomena, which are quite useful to the design and development of magnetic devices, sensors and actuators, magneto-electronics, and electrochemical and electromechanical systems. Some applications of nanoferrites are shown in Figure 1

Figure 1: Applications of nanosized ferrites [3]

A ferrite is a ceramic material that is made up of iron oxide (Fe_2O_4) in large proportions mixed with metallic elements such as cobalt (Co), manganese (Mn), nickel (Ni), zinc (Zn) in small proportions. Both the metal and the iron oxide have ferrimagnetic and electrically non-conductive properties. A ferrimagnetic material is one that retains spontaneous magnetization due to its unequal opposing magnetic moments. Hard and soft ferrites are the two general groups into which ferrites are divided. Due to their high coercivity, hard ferrites are challenging to magnetize. As a result, these materials are used to make permanent magnets that are used in high frequency applications, switch mode power supplies, dc-dc converters, microwave absorbing systems, washing machines, TVs, refrigerators, loudspeakers, and other devices [4-6]. On the other hand, soft ferrites can easily change their magnetization because of their low coercivity. Soft ferries are good conductors of magnetic field which has led to its wide range of applications in the electronic industry such as developing transformer cores, high frequency inductors and as microwave components .Soft ferrites also have low cost, high permeability, low loss, immediate and temperature stability, and high resistivity. The structural, electrical and magnetic properties are mainly dependent upon the substitution of metal elements in tetra- and/or octa-hedral sites of ferrite to design a specific application.

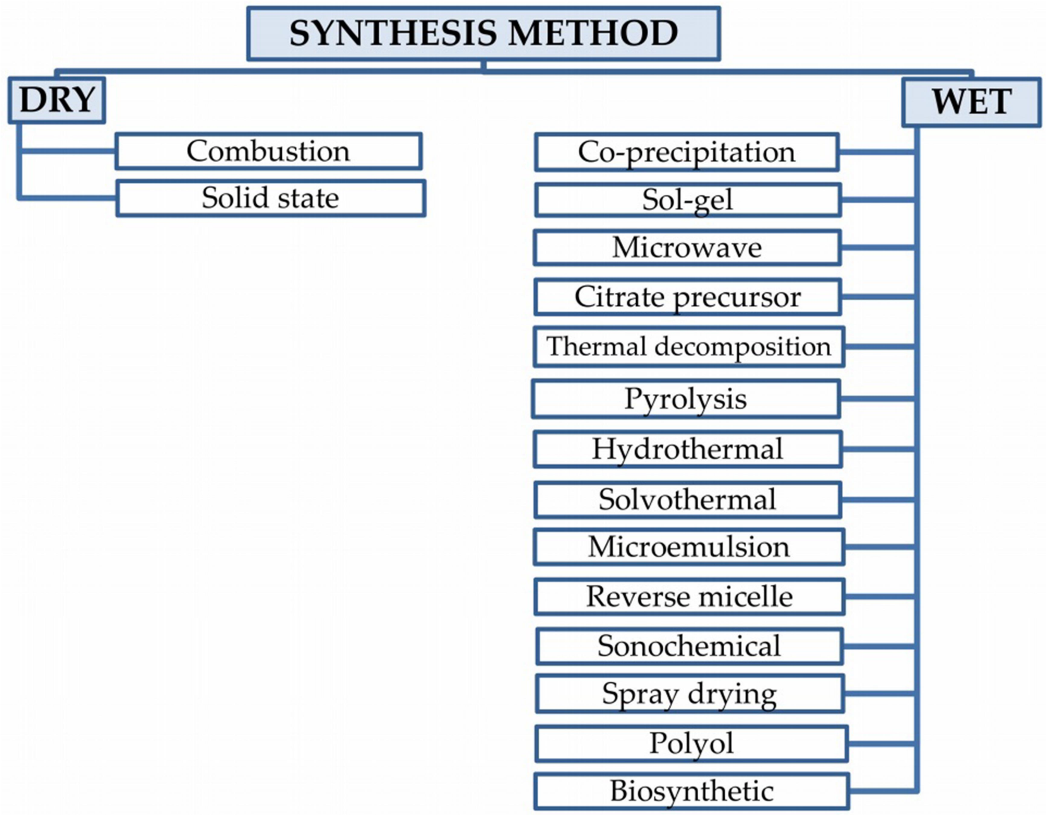
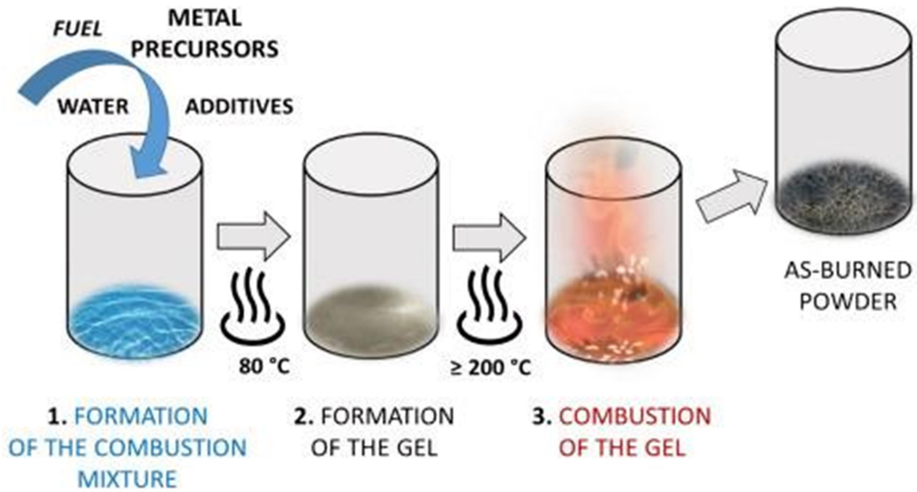


Figure 2. Synthesis methods to prepare ferrite particles [3]

The chemical and physical properties of transition metal nanoferrites (MFe_2O_4; M = Co, Ni, Zn, Mn, Cu) are dependent on the synthesis method and conditions. Therefore, choosing the right synthesis pathway is essential to customizing the characteristics and producing high-quality nanoferrites [1]. Numerous methods can be used to create nanosized ferrites (Figure 2), and the ability to create nearly any solid nanoferrite solution opens the door to customizing its characteristics for a variety of uses [7]. The ferrite synthesis methods are classified in three ways: (i) physical, chemical, and biological, depending on the processes involved; (ii) dry and wet methods, depending on whether a solution is present; and (iii) conventional and non-conventional, depending on their novelty. It is challenging to categorically group the synthesis procedures despite a number of classification techniques since occasionally distinct processes Comparative study of different methods of synthesis of Metal Oxides.

Combustion Method



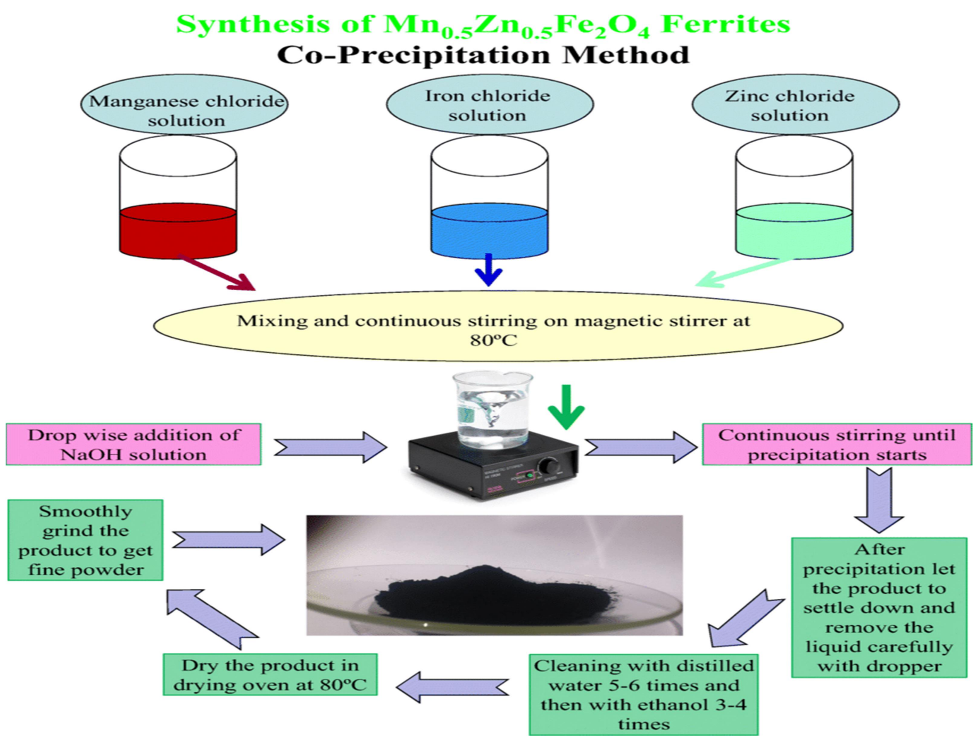
The combustion technique is an effective option for creating high-quality CoFe2O4, NiFe2O4, and MnFe2O4 nanoparticles. By adjusting the ratio of nitrates to fuel, it is possible to customize the size, magnetic saturation (MS), and hysteresis correction (HC) values [8,,9]. Nanocrystalline NiFe2O4 was successfully synthesized by combining metal nitrates with citrate to create a colloidal solution (sol), followed by continuous heating of the xerogel during the auto combustion process, leading to the formation of a loose powder, which was then annealed at 700 °C [10,,11]. This approach is time-efficient, yields pure and homogeneous nanoparticles without producing waste, although it does require high temperatures [12,13]. The excellent purity of materials produced through the combustion process is due to the elimination of undesirable impurities that become volatile at elevated temperatures.[14,15]

Applications of Metal Oxides Synthesized by Combustion

1. Catalysis: Metal oxides like TiO_2, CuO, and ZnO are widely used as catalysts in chemical reactions, including oxidation, hydrogenation, and pollution control (e.g., NOx reduction) [16]. Combustion-synthesized metal oxides often have high surface areas and catalytic activity, making them effective in catalytic processes.
2. Energy Storage and Conversion: Metal oxide materials such as $LiCoO_2$, MnO_2, and Fe_2O_3 are key components in batteries (e.g., lithium-ion batteries), supercapacitors, and fuel cells [17]. These metal oxides are valued for their high energy density and stability in electrochemical systems.
3. Environmental Remediation: Metal oxides like TiO_2 are used in photocatalysis for the degradation of organic pollutants and in water splitting for hydrogen production. Combustion-synthesized TiO_2, in particular, is known for its high photocatalytic efficiency [18]

4. Ceramics and Sensors: Metal oxides such as alumina (Al_2O_3), zirconia (ZrO_2), and tin oxide (SnO_2) are used in advanced ceramics and gas sensors. Combustion-synthesized metal oxides have improved purity and fine particle sizes, which are beneficial for these applications [19].

Co-Precipitation Method



The co-precipitation method is among the most commonly employed techniques due to its high yield and straightforwardness in generating high-purity ultrafine magnetic nanostructured ferrites [22,23]. This approach is quite simple and economical, allowing for easy control over particle size and composition, operating at low temperatures, and resulting in materials with excellent crystallinity, uniformity, and favorable textural properties [1,22]. However, the primary limitations of co-precipitation methods include significant agglomeration, subpar crystallinity, a wide range of particle size distribution, and the need for precise pH management. [24,25,26]. In this scenario, homogeneous solutions are created by dissolving inorganic salts, such as chloride, sulfate, and nitrate, in water or other solvents. Following the adjustment of pH to a level between 7 and 12 while maintaining continuous stirring, the precipitate is gathered through filtration or centrifugation, then washed and dried. The rate of pH change leads to the aggregation of particles and the growth of crystals. [22]. The most common way to synthesize nanostructured ferrites by chemical co-precipitation method is using Co^{2+} and Fe^{3+}

salts in the presence of a strong base [20]. By adjusting the experimental parameters (i.e. reaction temperature and time, reagents feeding rate and concentration, pH, drying temperature, etc.), the size, shape, and magnetic properties of the nanostructures may be controlled [27].

CoFe2O4 NPs (2–47 nm) were synthesized from metal chloride salts using different concentrations of aqueous NaOH and NH4OH solution using a reaction time of 2 h and variable reaction temperature (20–100 °C).
To carry out the precipitation reaction, an aqueous solution of metal chloride was slowly added to a preheated boiling aqueous alkali solution .The size of the crystallites depends on the reaction temperature, time, concentration of the main solution, and pH [27].

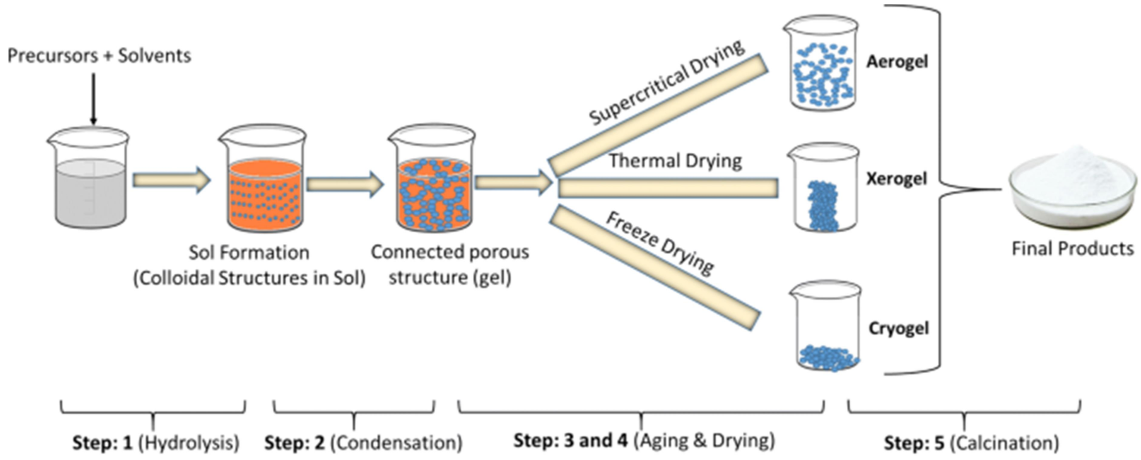
Another study reported the preparation of $CoFe_2O_4$ NPs (2–14 nm) by controlling the co-precipitation temperature of Co^{2+} and Fe^{3+} ions in alkaline solution from 20 to 80 °C [12]. The co-precipitation of $CoFe_2O_4$ nanocrystals by mixing Fe^{2+}, Co^{2+} and NaOH in the presence of oxidizing agent such as KNO_3 to convert Fe^{2+} and Fe^{3+} was reported by Chia et al. [28] and Senapati et al. [29]. However, due to the coexistence of both Fe^{2+} and Fe^{3+} ions, besides $CoFe_2O_4$, a secondary phase such as magnetite (Fe_3O_4) is also formed [28]. Moreover, the Hc values of particles obtained by this method are not very high and depend on the amount of surfactant used and the annealing temperature, which promotes a stable colloidal dispersion of NPs [29].

Applications of Metal Oxides Synthesized by Co-Precipitation

1. Catalysis: Co-precipitation is often used to prepare metal oxide catalysts, such as iron oxide (Fe_2O_3), zinc oxide (ZnO), or mixed-metal oxides. These materials are used in various catalytic reactions, including oxidation, reduction, and hydrogenation reactions, as well as in pollution control (e.g., NOx reduction) [30]
2. Energy Storage: Co-precipitated metal oxides like manganese oxide (MnO_2) and cobalt oxide (Co_3O_4) are used in energy storage devices such as supercapacitors and lithium-ion batteries due to their high surface area, conductivity, and electrochemical properties [31].
3. Sensors: Metal oxide semiconductors such as SnO_2, TiO_2, and ZnO, synthesized by co-precipitation, are widely used in gas sensors, particularly for detecting gases like CO, NO_2, and ammonia [32]. These sensors operate based on changes in the electrical conductivity of the metal oxide when exposed to different gases.
4. Photocatalysis: Co-precipitated metal oxides, particularly TiO_2, are widely used in photocatalysis for environmental applications such as the degradation of organic pollutants and water splitting for hydrogen production [33].

5. Pigments and Ceramics: Metal oxides produced by co-precipitation, such as TiO_2 and ZnO, are also used in the production of pigments, ceramics, and coatings due to their stability, high temperature resistance, and aesthetic properties.

Sol-Gel method



The sol-gel method is a low-temperature process based on the hydrolysis and condensation reactions of metal precursors (salts or alkoxides), leading to the formation of a three-dimensional inorganic network [34,38].

Sols are obtained by converting monomers into colloidal solutions, while gels are obtained after evaporation of the solvent by binding particles into a network [22].

The sol-gel method is a simple, inexpensive and environmentally friendly method for reparing nanocomposites (NCs) because it provides good control over the microstructure, particle size, dispersion, structure and nanocomposites (NCs). chemical composition by carefully monitoring the preparation parameters [22,35,36]. Using the sol-gel method, very fine, dense, uniform single-phase ferrite nanoparticles were prepared. Compared to other conventional methods, sol-gel exhibits good stoichiometric control and also allows the production of ferrite at relatively low temperatures [8].

The resulting nanomaterials can be formed as films or colloidal powders [22].

The main drawback is that the effectiveness and period of synthesis processes are limited.[22] The average particle size of 27 nm NAFE2O4 nano structures were synthesized using glycolic acid as a health agent. In the case of COFE2O4 @ sio2, the Sio2 network protects NP and minimizes surface roughness and back disorder.

HC values at room temperature (RT) for COFE2O4 @ sio2 were much higher than those of unpaid COFE2O4 [35, 39].

Bottom-up approach via sol-gel, micro emulsion, hydrothermal, chemical co-precipitation or top-down approach like ball milling and high temperature sintering.

Although the chemical routes such as sol-gel route yields more promising results in the synthesis of nanoferrites, several preparation conditions such as dilution, fuel/oxidant ratio, pH and temperature can Affects the formation of ferrite and its properties. The proposed research work aims to systematically study the effect of substitution of metal ions in the parent ferrite system. Moreover, the effect of varying parameters during synthesis of ferrites on the structural and magnetic properties of ferrite material will be investigated.

Considering the factors that i) substitution of metal ions and ii) synthesis method greatly affect the structural, chemical and magnetic properties of parent ferrite, it is decided to systematically investigate the above mentioned two factors.

Applications of Metal Oxides Synthesized by Sol-Gel

1. Catalysis: Metal oxide catalysts, such as TiO_2, ZrO_2, and CuO, are commonly synthesized using the sol-gel method. These catalysts are widely used in reactions like oxidation, hydrogenation, and in environmental applications such as photocatalytic degradation of pollutants [32]. The sol-gel method allows for the preparation of catalysts with high surface area and controlled porosity, which are essential for improving catalytic activity.
2. Energy Storage: Metal oxide materials such as MnO_2, Co_3O_4, and NiO, which are used in lithium-ion batteries, supercapacitors, and fuel cells, can be synthesized through the sol-gel method. The sol-gel process enables precise control over particle size, morphology, and porosity, which are important for enhancing the performance of these materials in energy storage devices [31].
3. Sensors: Metal oxide semiconductors such as SnO_2, TiO_2, and ZnO, produced by sol-gel, are widely used in gas sensing applications. The controlled microstructure and high surface area of

sol-gel-derived oxides enhance their sensitivity to gases like NO_2, CO, and alcohols, making them suitable for environmental monitoring and safety applications [32].

4. Photocatalysis and Environmental Remediation: The sol-gel method is particularly useful for synthesizing metal oxide photocatalysts, such as TiO_2, which are employed in environmental applications like water treatment, air purification, and photocatalytic hydrogen production [33]. These materials exhibit excellent photocatalytic properties due to their high surface area and controlled morphology.
5. Optical and Ceramic Coatings: Sol-gel-derived metal oxides such as SiO_2, TiO_2, and Al_2O_3 are widely used for optical coatings, anti-reflective coatings, and protective coatings. These materials exhibit high transparency, hardness, and stability, making them suitable for use in a variety of optical and electronic devices [30].

Merits and Demerits of Magnetic Oxides by different Methods

Magnetic oxide nanoparticles are widely synthesized using various methods, each with its unique advantages and challenges. Below is a comparison of the **Sol-Gel Method**, **Co-precipitation Method**, and **Combustion Method** in terms of their merits and demerits:

Synthesis Methods	**Merits**	**Demerits**
Combustion Method	1)Self propagating an effective low cost method. 2)An exothermic reaction occurs, potentially saving energy and time. 3)low energy input. 4)Use of relatively simple equipment 5).Use of relatively cheap reactant(like nitrates) 6)Formation of high purity product with a variety of size and shape.	high temperature synthesis. The presence of a relatively large amount of carbon in the final product.
Co-precipitation Method	1)High-Yield Production: Coprecipitation is widely used due to its high-yield production of nanoparticles. It allows for the efficient synthesis of metal oxide nanoparticles. 2)Simplicity and Low Cost: The method is straightforward and cost-effective. It doesn't require complex equipment or elaborate procedures. 3)Eco-Friendly Reaction Conditions: Coprecipitation can be carried out under mild and environmentally friendly conditions, minimizing the use of hazardous reagents. high product yield	1)Low Reproducibility: Despite its advantages, coprecipitation suffers from low reproducibility. Variability in particle size and composition can occur. 2)Product Separation: After precipitation, the resulting product needs to be separated from the solution. This step can be cumbersome. 3)Salt-Containing Solutions: Coprecipitation generates large volumes of salt-containing solutions, which can be challenging to handle and dispose of.

Sol-gel method	1)Cost-Effective: The sol-gel process is relatively inexpensive, making it an attractive choice for large-scale production of metal oxides . 2)Control Over Composition: It allows precise control over the chemical composition of the resulting materials. This is especially beneficial for doping or adding transition metals . 3)Low Reaction Temperature: The sol-gel method operates at lower temperatures compared to some other preparation techniques . 4)High Yield: It offers high yield, ensuring efficient material production . 5)Homogeneity: The produced material exhibits homogeneity, which is advantageous for consistent properties . 6)Formation of Complex Structures: Sol-gel synthesis can lead to the formation of complex structures or composite materials	1)Longer Processing Time: The sol-gel process can be slower compared to some alternative methods the sol-gel method offers cost-effectiveness, compositional control, and versatility, but it requires careful consideration of drying methods and is primarily applicable to metal oxides. Researchers continue to explore its potential in various fields, including optics, electronics, and energy applications 2)Drying Process Dependency: The properties of the dried gel significantly depend on the drying method used. Different applications require specific drying techniques.

Conclusion

Sol-gel method is one of the best synthesis method for metal oxides. This paper presented survey of different methods for synthesis of metal oxides.

Few of them are Combustion, Co-precipitation, Thermal decomposition, Sol-gel etc.

It is also concluded that Sol-gel method has potential control of the whole reaction during synthesis. It is more efficient method due to high purity of the product, the narrow particle size distribution, and the achievement of uniform nanostructure at low temperatures.

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