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Brief Review on the Effect of Synthesis Method and Doping on the Structure and Magnetic Properties of Zinc Ferrites

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ABSTRACT

In this paper, various synthesis methods of zinc ferrites have been reviewed and the effect of the synthesis method and doping, on the structure and magnetic properties of zinc ferrites were studied. Ferrites have used extensively in different fields due to their higher magnetic strength and larger electrical resistance. Reduction in the size of these materials to the nanoscale has expanded their domain of applications. Zinc ferrites remain one of the widely studied materials because of their unique properties such as chemical and thermal stability, and the particle size dependence of magnetic properties. The synthesis procedures and precursor materials were decided the size, structure, shape, and magnetic strength of these materials. The magnetic properties of nano ferrites depend on the particle size and the preparation route.

Keywords: Zinc ferrites, Synthesis methods, Microscopy, Particle size, saturation magnetisation.

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I. INTRODUCTION

Ferrites are generally crystallized in the form of AB_2O_4 with A representing tetrahedral site and B octahedral site. A site is normally occupied by a divalent cation leading to the formation of spinel structure. These cations can be distributed among both A and B sites which can lead to the formation of inverse spinel or mixed spinel structures [1-3]. Lodestone, or magnetite, was a naturally occurring iron ferrite with chemical formula Fe_3O_4 in which A site and B sites are occupied by the divalent and trivalent ferrous ions [4,5]. The structure of $ZnFe_2O_4$ in the bulk compound is normal spinel ferrite. The diamagnetic Zn^{2+} ions occupancy of the tetrahedral A-sites renders all Fe^{3+} ions on the octahedral B-sites[6-9].Moreover, $ZnFe_2O_4$ is a soft magnetic oxide due to the nonmagnetic properties of Zn^{2+}ions. Among the spinel ferrites, zinc ferrite has a special kind because of its unique properties such as chemical and thermal stability, and the particle size dependence of magnetic properties. Zinc ferrites show higher saturation magnetization and magnetic susceptibility leading to the potential applications in the field of magnetic resonance imaging (MRI)[10], photoelectrochemical cells[11], photochemical hydrogen production [12], gas sensors [13], photocatalysis [14], ferrofluids [15], dye-sensitized solar cell [16], and information storage[17].

Altering the properties of materials could be achieved by a reduction in the sizeof the materials to the nanoscale which widens the scope of application of mostof the materials [18]. The properties of nanophase materials were decided by the synthesis methods, dopant atoms, size, and morphology of the particles. Over the years of extensive investigations, it has been manifested that the synthesis technique dictates the starting precursor materials for ferrite preparation which will define the ease or difficulty in the synthesis procedure [19]. Also, the formation of the pure or impure phase of zinc ferrites was found to be dependent on the synthesis techniques adopted. It has been shown that the substitution of dopant, the parent spinel ferrite with various metal ions leads to structural disorder and lattice strain, thereby improving the electrical and magnetic parameters. Size reduction has been found to show variation in Neel temperature, coercivity, saturation magnetization etc. This review is an attempt to consolidate various synthesis methods along with the variety of precursors used for each method and the properties obtained employing different characterization techniques for pure and doped Zinc ferrites. Comparison has been done in various possible ways to identify the quality of samples obtained through various methods and for various applications.

II. SYNTHESIS METHODS

Structurally perfect pure and doped $ZnFe_2O_4$ nanocrystals were successfully prepared by various methods like auto-combustion[20], Sol-gel auto-combustion [21], Co-precipitation [22-24], sol-gel [25], Hydrothermal[26-28], Solid-state reaction [29], Thermal decomposition[30], Solvothermal method[31], Sonochemical technique[32], Microwave combustion method[33], High energy ball mill method [34], Standard ceramic technique [35], Microwave-assisted ball milling[36], etc. It has been found that every method has its own merits and demerits.

2.1 Auto-combustion

This process is convenient, inexpensive, environmentally friendly and efficient for the preparation of zinc ferrite nanoparticles. In this technique, the stoichiometric amount of precursor salts and urea were mixed in an agate mortar. Urea was added to the mixture as fuel, and the mixture was transferred to a quartz crucible. A reaction occurs within the mixture leads to combustion which gets completed in a very short time of 3 to 5 minutes. Foamy and highly porous precursor mass was obtained which was collected and then powdered. The ferrite powder was pre-sintered, pelletized and then sintered again at higher temperatures to yield pure zinc ferrites. The pre-sintering and sintering temperatures again depend on the composition and precursor material chosen for particular cases.

2.2 Sol-gel

Good control of composition, purity, homogeneity, particle sizes and distribution can be obtained using the sol-gel method. The chemical reagents used in the preparation are zinc nitrate, ferric nitrate, urea, and citric acid. An appropriate amount of zinc nitrate and iron nitrate along with citric acid was first dissolved in a medium like of urea solution as a catalyst, and then a small quantity of distilled water was added to this mixture. This mixture was subjected to vigorous stirring, and the resulting solution was allowed to evaporate to form a gel. Heating and drying of this gel at different temperatures in a furnace, yielding nanocrystalline $ZnFe_2O_4$ material.

2.3 Sol-gel auto-combustion

In this method, the appropriate amounts of nitrates and urea/citric acid/ thiourea as fuel was first dissolved into deionized water to form a mixed solution. The pH value of the solution was adjusted to about 9.5 using ammonia. Then, the mixed solution was poured into a dish and heated at 70-100^o C under constant stirring to transform into a dried gel. Being ignited, the dried gel burnt in a self-propagating combustion way to form a loose powder. This process was continued for 3 to 4 hours, after which the transparent solution turned to a viscous brown gel followed by foaming of the gel. The foamy gel was kept on a preheated oven at 350°C which caused its spontaneous ignition. The combustion reaction was completed within a few seconds and loose powder was formed. This was crushed and ground thoroughly. The puffy, porous brown powders as-combusted was calcined at the temperature of 750–1000˚C for 4h with a heating rate of 10˚C/min.

2.4 Co-precipitation

The metal ion solution was prepared by adding chlorides of zinc and ferric into distilled and deionized water. A one molar solution of NaOH was prepared separately in deionized water. The solution containing the metal ions was mixed dropwise into the NaOH solution with rigorous mechanical stirring on a magnetic stirrer. The solution thus formed kept under isothermal static conditions in a preheated water bath for digestion treatment. The precipitates were filtered and washed with deionized water and dried in an oven at 60◦c and the powders were pressed into pellets.

2.5 Hydrothermal

An appropriate amount of metal nitrates was first dissolved in 40 ml deionized water. Then 2 ml of ethyl amine was added to the solution by dropwise under the magnetic string at room temperature. Finally, we obtained precipitated solution. The mixture was continuously stirred for half an hour and transferred to 50 ml Teflon-lined autoclaves. The autoclave was sealed and maintained at 160 °C for 15 h then allowed it to reach the room temperature. Finally, the brown precipitates were collected by centrifugation using deionized water and absolute ethanol washed three times and dried at 60 °C for 2 h at hot air oven. Then the product was crushed by mortar and annealed in an air atmosphere at 300°C and 600°C for 10h yielding $ZnFe_2O_4$.

2.6 Microwave combustion method

Zinc nitrate, ferric nitrate and urea were used as a fuel for this reaction. For the preparation of zinc ferrite using the microwave technique, the precursor's mixture in urea was placed into a microwave oven and exposed to the microwave energy in a 2.45GHz multimode cavity at 750W for a few minutes. The reaction in the microwave cavity was designed since urea has a high dielectric loss value, which will be heated very fast in the microwave heating system. After completion of the reaction, the solid powder was obtained and then washed with ethanol and dried at 70oC for1h yields the zinc ferrite particles.

2.7 Solvothermal method

An appropriate amount of ferric chloride and zinc nitrate were dissolved in the dispersion. Then, 3.6g of NaAc, 0.8 ml of polyethene glycol 200 (PEG 200) were added to the dispersion under vigorous stirring at room temperature for 30 minutes. Subsequently, the mixture was sealed in a Teflon-lined stainless-steel autoclave and maintained at 200 ◦C for 12 h. Then, the mixture was cooled to ambient temperature. The brown product was collected by a magnet and rinsed with deionized water and ethanol until there were no chloride ions in the solution. The brown final product was dried in vacuum at 60◦C for 12h.

III. STRUCTURAL AND MAGNETIC PROPERTIES

3.1 Pure Zinc Ferrites

Extensive work has been done to establish relationships between particle size, structure, surface area, and shape of pure and doped zinc ferrite nanoparticles. As stated earlier, the properties of these materials are also sensitive to the preparation conditions and methods adopted. It has been found that even a single step variation affects structural properties leading to changes in various other properties. Table 1, summarizes the crystalline size variations and saturation magnetization depending on different synthesis methods adopted.

Identification of suitable sintering temperatures, raw materials and preparation technique decides the possibility of achieving phase pure $ZnFe_2O_4$. It has been found that in the co-precipitation technique, pure phase zinc ferrites could be achieved at low temperature if chlorides were used as precursor materials. On the other hand, if nitrates were used as precursor materials, then higher annealing temperatures becomes mandatory for the formation of pure phase [37].

The crystalline size and saturation magnetization are depending upon the synthesis method, precursors materials, temperature, and fuel agents in a particular synthesis method. Reported values have been shown that the maximum value of saturation magnetization clearly depends on a specific synthesis route. The observed structure for the sample in the hydrothermal method is more crystalline than the sample in the co-precipitation method. The reported data reveals that the lowest crystalline size is achieved by the co-precipitation method with a low precipitation temperature.

Table 1 Reported Values of crystalsize, Saturation Magnetization for pure Zinc ferrite Nanoparticles Prepared by Various Methods

S.No	Synthesis Method	Sintering/ calcination temperature (^{O}C)	crystal size (nm)	Lattice constant (A^{o})	Saturation Magnetization Ms (emu*g-1)	Ref
1	Sol gel auto combustion	750 – 1000	17			21
2	Co precipitation	20-80	5-10	8.45-8.47	7.3	22
3	Sol gel (urea)	600-1000	29-52	8.347-8.410	3	25
4	Sol gel (citric acid)	400-1000	18-42	8.361-8.379	1.9	25
5	Hydrothermal	300 - 600	21-28	8.412	9.10 - 12	26
6	Hydrothermal synthesis		300	8.435	54.64	27
7	Thermal decomposition	290	9.8	8.411	65.4	30
8	Sonochemical	550	6 - 17			32
9	Ball milling	1000	10 - 18.9	8.4183 – 8.4274		34

10	Microwave assisted Ball milling	<100	23.24-26.19		82.23	36
11	Starch assisted Sol gel auto combustion	600	8.72	8.4443	2.77	38
12	Ball milling		11-14		10-75	39

A short analysis has been done in the following section concentrating on the sol-gel technique. Table 2 showsthe precursors materials for various fabrication methods.

Table 2. The precursors materials for various synthesis methods

S No	**Synthesis Method**	**Precursors Materials**	**Ref**
1	Sol gel Auto combustion	Zn (NO3)2·4H2O, Fe (NO3)3·9H2O and Thiourea (CS (NH2)2	21
2	Co precipitation	Iron (III)chloride hexahydrate (FeCl3.6H2O), zinc (II)chloride (ZnCl2), sodium hydroxide (NaOH) and acetone	22
3	Sol-gel	Zn (NO3)2.6H2O, Fe (NO3)39H2O and urea	25
4	Sol-gel	Zn (NO3)2.6H2O, Fe (NO3)39H2O and citric acid	25
6	Hydrothermal	Zinc nitrate hexahydrate [Zn (NO3)2 · 6H2O], ferric nitrate nonahydrate [Fe (NO3)3 · 9H2O], and ethylamine (C2H7N)	26
5	Thermal decomposition	ZnAc2.2H2O, oleic acid, Fe (CO)5 octyl ether, Anhydrous ethanol (C2H5OH, chromatogram grade)	30
6	Starch assisted Sol gel Auto combustion	Zn (NO3)2·4H2O, Fe (NO3)3·9H2O, and aqueous solution of starch (C6H10O5) n	38
7	Sol-gel	Fe (NO3)3·9H2O, Zn (NO3)2·4H2O and Ethylene glycol C2H6O2	40

The precursor materials also significantly affect the crystalline size and magnetic properties. It has been shown that for the same preparation technique with different precursor materials the crystalline size is different and their magnetic properties also different. The crystalline size of zinc ferrite prepared by sol-gel method with various fuel agents as precursors has various crystalline sizes. The crystalline size in citric acid fuel agent is relatively smaller than the particle size in urea fuel agent. Nanoparticle prepared in the same method with different fuel agents exhibits different magnetic properties [25]. In literature for the preparation of manganese ferrites, precursors materials in addition to metal nitrates and metal chlorides, the metal sulphates were also used [41]. But the zinc ferrite preparation zinc sulphates were not preferred as a precursor material.

3.2 Doped Zinc Ferrites

Various synthesis methods with various dopants forvarious concentrations and the related parameters have been given for zinc ferrites.

3.2.1 Cobalt Doped Zinc Ferrites

Cobalt doped zinc ferrites are prepared by various methods as given below in table 3. As the concentration of cobalt increases the particle size is increasing and lattice parameter decreases. The particle size is a function of sintering temperature. As the cobalt concentration increases its saturation magnetisation also increases. The ferrites exhibiting superparamagnetic behaviour for lower concentrations of cobalt and the ferromagnetic behaviour for higher concentrations of cobalt. In all the reported values with different dopants, the cobalt doping is much enhancing the saturation magnetisation that is the cobalt doping improves its magnetic property significantly. Even in nanoscale, the cobalt doped zinc ferrites have lower saturation magnetisation as compared to its bulk counterpart (MS=80emu*g^{-1}) [42]. Due to change of cation distribution in nanoscale, the sample prepared with Starch assisted sol-gel auto combustion method exhibiting ferrimagnetic behaviour, and bulk sample shows paramagnetic behaviour.

Table 3
Reported Values of crystal size, Saturation Magnetization for cobalt doped Zinc ferrite Nanoparticles Prepared by Various Methods

S.No	Synthesis Method	Cobalt Concentration (%)	crystal size (nm)	Lattice constant (A°)	Saturation Magnetization M_s (emu*g^{-1})	Ref
1	Microwave Auto combustion	0-0.5	43-49	8.224 – 8.4438	1.638 - 65.20	33
2	Microwave absorber	0.6	45	-------	92.3	47
3	Co precipitation	0-0.5	37-51	8.407 – 8.439	2.6 - 82	48
4	ChemicalCo-precipitation	0.2	□20	-------	32	49
5	Precipitation and Hydrothermal	0-0.4	8.3 – 11.4	8.431 – 8.454	40 - 98	50
6	Sol-gel	0.5- 0.7	18 - 49	8.411-8.346	29.7 (0.5)	51
7	Microwave combustion	0 – 0.5	25.69 – 35.68	8.387 – 8.407	31.71 – 73.80	52
8	Mechanochemical method	0.2	□20		30	53

3.2.3 Nickel Doped Zinc Ferrites

Doping of nickel has promoted the growth of the crystalline size, resulting in a decrease of lattice strain. It was observed that irrespective of the synthesis method lattice parameter decreases with the increase of Ni2+ ion concentration. Nickel ferrites have a resonant frequency in a high frequency; therefore, they are more useful, especially in microwaves. For high sintering temperature, the fine-grained microstructural feature may possess good frequency stability and high quality. The uniformly spherical Ni-doped zinc ferrites with different particle size can be obtained with different annealing temperatures. In microwave combustion method the particle size and saturation magnetization are significantly different for the method in which urea as a fuel precursor compared to glycine fuel precursor [43,44].

Table 4
Reported Values of crystal size, Saturation Magnetization for Nickel doped Zinc ferrite Nanoparticles Prepared by Various Methods

S.No	Synthesis Method	Nickel Concentration (%)	crystal size (nm)	Lattice constant (A°)	Saturation Magnetization Ms (emu*g^{-1})	Ref
1	Microwave combustion (Urea)	0 - 0.5	41.2 -45.94		1.681 - 57.89	43
2	Microwave combustion (Glycine)	0 - 1	32 - 42	8.4056 - 8.3322	47.68- 40.71	44
3	Auto combustion (Citric acid)	0.2 - 0.8	8.3-29.8	8.393 - 8.3718		54
4	Coprecipitation	0.5 - 0.7	10.6 - 12.3	8.381 - 8.346		55
5	Sol-gelAuto combustion	0.7	35	8.38	69.1	56
6	Sol-gel	0 -1	12 - 26	.405 - 8.334	13 - 15	57

3.2.4 Manganese Doped Zinc Ferrites

The particle size of Manganese doped zinc ferrites have relatively smaller than the other dopant zinc ferrites. The particle size is increasing with increasing manganese concentration in all methods. Except for chemical precipitation method in all the methods, the lattice constant is increasing with increasing of manganese concentration because of due to its radius which is more than the zinc radius.

Table 5
Reported Values of crystal size, Saturation Magnetization for Manganese doped Zinc ferrite Nanoparticles Prepared by Various Methods

S.No	Synthesis Method	Manganese Concentration (%)	crystal size (nm)	Lattice constant (A^o)	Saturation Magnetization Ms (emu*g^{-1})	Ref
1	Auto combustion	0.4	12	8.44		46
2	Chemical Co precipitation	0.3 - 0.7	8.7 - 14.5	8.398 - 8.447	29.24 - 46.66	58
3	Double sintering	0.66 -0.99		8.46 - 8.51		59
4	Chemical coprecipitation	0 - 0.5	4.62- 6.84	8.451- 8.396		60
5	Sonication Assisted microwave irradiation	0 - 0.5	8.55- 9.39	8.384 - 8.448	3.17- 51.73	61
6	Spray pyrolysis	0.5	7			62

3.2.5 Magnesium Doped Zinc Ferrites

Substitution of magnesium in zinc ferrites significantly changing properties of zinc ferrites. With the increase of concentration of magnesium, the crystalline size and lattice constant was decreased because of the relatively smaller radius of magnesium ion than zinc ion.

Table 6
Reported Values of crystal size, Saturation Magnetization for magnesium doped Zinc ferrite Nanoparticles Prepared by Various Methods

S.No	Synthesis Method	Magnesium Concentration (%)	crystal size (nm)	Lattice constant (A^o)	Saturation magnetization Ms(emu*g^{-1})	Ref
1	Chemical route	0.2 - 0.8	48.17 - 46.74	8.191 - 8.115		63
2	Co precipitation	0 - 0.5	31 - 21	8.432 - 8.409		64
3	Solgel	0 - 1	18.65 - 9.70	8.433 - 8.336		65
4	Sol gel Auto combustion	0.2 - 0.6	36.54 - 17.78	8.449 - 8.444		66
5	Rapid Microwave combustion	0 - 0.8	41.20 - 15.87	8.443 - 8.427	1.638- 64.98	67

3.2.6 Cadmium Doped Zinc Ferrites

Cadmium substitution in zinc ferrites was significantly changing the structure of the crystal. It was observed that with the increase of cadmium concentration the particle size is decreasing and lattice constant is increasing unlikely other dopants. Compared to all the reported values of cadmium doped zinc ferrites have relatively lesser saturation magnetization.

Table 7
Reported Values of crystal size, Saturation Magnetization for cadmium doped Zinc ferrite Nanoparticles Prepared by Various Methods.

S.No	Synthesis Method	Cd Concentration (%)	crystal size (nm)	Lattice constant (A^o)	Saturation Magnetisation (emu*g-1)	Ref
1	Microwave assisted Auto combustion	0 - 0.5	42 - 12	8.4434 - 8.512	3.174- 1.546	68
2	Soft Chemical synthesis	0 - 1	55 - 49	8.44 - 8.69		69

3.2.7 Copper Doped Zinc Ferrites

The reported values were showing that particle size in microwave combustion method is relatively higher than other methods. It is an interesting fact that observed in sonication assisted microwave method the particle size is decreasing with increasing of copper concentration because of due to strong bond energy between copper and oxygen compared to zinc and oxygen [45].

Table 8
Reported Values of crystal size, Saturation Magnetization for copper doped Zinc ferrite Nanoparticles Prepared by Various Methods.

S.No	Synthesis Method	Copper Concentration (%)	crystal size (nm)	Lattice constant (A^o)	Saturation Magnetisation (emu*g-1)	Ref
1	Sonication assisted microwave method	0-0.5	8.55 – 5.84	8.384 – 8.33	16 – 74.1	45
2	Microwave Combustion	0 - 0.5	41.20 - 45.84	8.443 - 8.413	1.638 - 58.58	70
3	Co precipitation	0.2	12.3			71

IV. CHARACTERIZATION

The structure and phase of the sample were confirmed by XRD techniques. The particle size is calculated by using XRD peaks with Scherrer formula. In various synthesis methods, the values of the crystallite size obtained by Scherrer's formula is different from those obtained by the Rietveld analysis. This is because of the effect of the internal strain, in that particular synthesis route. As reported values show that the particle size measured by XRD technique is different for different synthesis routes. The degree of crystallinity and particle size depends upon various experimental parameters in synthesis routes. XRD pattern reveals that the degree of crystallinity increases with the precipitation temperature.

Scanning Electron microscopy gives the information regarding surface morphology normally shape and size of the particles. It has been reported that the shape and particle size are different in different synthesis methods. The particles synthesized by the co-precipitation method the surface has compacted shapes with round particles, while for particles obtained by the hydrothermal method the particles exhibit elongated shapes. The particle shape is spherical in pure ZnFe2O4 as prepared by sol-gel auto combustion method. Even with doping also in some ferrites, the spherical and uniform particles were formed. The particles have a spherical shape at a low annealing temperature and hexagonal shape at higher annealing temperature.

The Transmission electron microscopy gives information regarding shape and particle size. In most of the synthesis methods as prepared particles are spherical shape and exhibit chain-like clusters and the particle size distribution is very narrow. Manganese substituted zinc ferrites prepared by auto-combustion the method shows the polycrystalline nature of spherical and hexagonal shaped particles [46]. The particle size and shape measured by TEM is different in various synthesis methods for pure and doped ferrites.

Saturation magnetization was measured at room temperature by using vibrating sample magnetometer. The maximum magnetisation is depending upon the synthesis route and also the dopant. The maximum magnetisation achieved by zinc ferrite prepared by the hydrothermal method is relatively higher than the zinc ferrites prepared by co-precipitation method. Saturation magnetisation values achieved in various synthesis methods are different. The reported values have shown that the maximum magnetisation was obtained in the microwave absorber method with cadmium doping [47].

V. CONCLUSION

Pure and doped zinc ferrite nanoparticles were obtained by various synthesis methods that governed the size, shape and structure of the final particles. The benefits or difficulties in the formation of ferrite nanoparticles is determined by the precursor materials, and the consequent synthesis technique to be adopted for various materials. Samples annealed at higher temperature were invariably found to lead to impure phases in these materials. This review confined itself to the synthesis Strategies and conditions adopted for the formation of pure and doped zinc ferrite samples. XRD, Microscopic techniques and vibrating sample magnetometer were discussed to understand the different structures and magnetic properties obtained supported the synthesis conditions and procedures.

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