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## Synthesis, Characterizations, and Magnetic Properties of Mixed Spinel $Mg_{1-x}Zn_xFe_2O_4$ Ferrites

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### Abstract

Mixed spinel  $Mg_{1-x}Zn_xFe_2O_4$  ferrites (where  $x = 0, 0.2, 0.4,$  and  $0.6$ ) nanoparticles were synthesized by using microwave-assisted combustion route. As-synthesized powdered samples were checked by XRD analysis, field emission-scanning electron microscopy, and vibration sample magnetometer to investigate the structural, morphology, and magnetic properties, respectively. XRD results exhibited that the crystallite size increases with the decrease of  $Zn^{+2}$  ion concentration for series of mixed spinel  $Mg_{1-x}Zn_xFe_2O_4$  ferrite expect  $x=0.2$ . All the mixed spinel  $Mg_{1-x}Zn_xFe_2O_4$  ferrite has different grain sizes with uniform distribution also presence voids in the samples. Pure magnesium ferrite has a lower net magnetization value but when magnesium ions ( $Mg^{+2}$ ) are replaced by zinc ions ( $Zn^{+2}$ ) then the value of saturation magnetization increases.

**Keywords:** Mixed spinel ferrites, Microwave, Glycine, Nanoparticles, Magnetic properties

### التصنيع والتوصيف والخصائص المغناطيسية لفرايتات السبينيل المختلط $Mg_{1-x}Zn_xFe_2O_4$

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### الخلاصة

تم تصنيع جسيمات نانوية من فرايتات السبينيل المختلط  $Mg_{1-x}Zn_xFe_2O_4$  حيث  $x=0,0.2,0.4$  and  $0.6$ ) باستخدام طريقة الاحتراق بمساعدة الميكروويف. تم فحص عينات المسحوق المصنعة بواسطة تحليل الأشعة السينية والمجهر الإلكتروني الماسح للانبعاثات ومقياس المغناطيسية لعينة الاهتزاز للتحقق من الخصائص الهيكلية والتشكيلية والمغناطيسية على التوالي. أظهرت نتائج الأشعة السينية أن متوسط حجم البلورات يزداد مع انخفاض تركيز أيون الخارصين لسلسلة من فرايتات الإسبينيل المختلط  $Mg_{1-x}Zn_xFe_2O_4$  ما عدا  $x = 0.2$ . تمتلك جميع فرايتات الإسبينيل المختلط  $Mg_{1-x}Zn_xFe_2O_4$  أحجام حبيبات مختلفة ذات توزيع موحد مع وجود فجوات. تكون قيم المغناطيسية منخفضة لفرايت المغنيسيوم النقي و تزداد قيمة مغنطة التشبع عندما يتم استبدال أيونات المغنيسيوم ( $Mg^{+2}$ ) بأيونات الخارصين ( $Zn^{+2}$ )

### 1. Introduction

Generally, ferrites are compound of mixed oxides of iron and one or more other materials which have ferrimagnetic properties[1]. Ferrites are classified into four types depending upon the crystal structure, such a spinel, ortho, hexagonal, and garnet ferrites, each having its

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importance. Spinel-type ferrites are a group of iron oxides as the main constituent, and metal oxides[2]. Due to their unique magnetic, thermal, electrical, and catalytic properties for high-tech applications, mixed spinel ferrite nanoparticles (MSFNs) are the particular research topic for the researcher all over the world [3-5]. Soft ferrites have a cubic spinel structure consists of 8 units of the form of  $M^{2+}Fe_2^{3+}O_4$ , where 'M' is a divalent atom of radius between 0.80 Å to 1.0 Å, such as Mn, Mg, Cd, Fe, Zn, Cu, etc. Magnesium-Zinc ferrite is a soft mixed ferrite of magnesium-ferrite and zinc-ferrite. Magnesium occupies octahedral (B) sites and Zinc occupies tetrahedral (A) sites in the spinel-structure with unit of the form  $(Zn^{2+}_{1-x}Fe^{3+}_x)[Mg^{2+}_xFe^{3+}_{2-x}]O_4$ , The limiting case, ( $x=1$ ) where  $x$  is the degree of inversion is called normal spinel ferrite and the other limiting case, ( $x=0$ ) is called inverse spinel ferrite. For a random distribution  $x$  can have value in between 0 and 1 [6-8].

Mixed spinel ferrites is gaining much interest among researchers, Aswad and Mutlak[5] studied the effect of synthesized  $Co_{1-x}Cu_xFe_2O_4$ , through hydrothermal route on the growth of some pathogenic bacteria, Abdelhasan et al.[9] synthesized Co-Zn ferrite nano particles less than 50 nm by microwave combustion and hydrothermal methods as a biomedical application, Amar et al.[10] synthesized Zn-doped Co-ferrite ( $CoFe_{1.9}Zn_{0.1}O_4$ ) magnetic nanoparticles via sol-gel method as a magnetic catalyst, and Manikandan et al [11] were synthesized powdered Mg-doped  $ZnFe_2O_4$  nanoparticles with mean size in the range of (15nm – 43nm) by a microwave combustion route.

Here, we demonstrate the series of mixed spinel ferrite  $Mg_{1-x}Zn_xFe_2O_4$  where  $x = (0, 0.2, 0.4, \text{ and } 0.6)$  were synthesized via microwave combustion route that is usually performed in solution media. This method was chosen due to simplicity and short time reaction.

## 2. Experimental

High purity nitrate salts of (Zinc- Magnesium: Iron) [from HIMEDIA Co., India] were used as the raw materials to prepare mixed spinel ferrite nanoparticles with a molar ratio (1:2) by microwave combustion route technique. Each material was weighed separately in stoichiometric ratio and dissolved in a suitable quantity of de-ionized water then the solutions were mixed thoroughly using a magnetic stirrer for 20 minutes to complete dissolution until a red color slimy solution is obtained. Stoichiometrically amount of fuel (glycine) with the chemical formula ( $C_2H_5NO_2$ ) was added[Glycine to nitrate ratio 0.33] to the mixture under stirring. After 20 minutes, a dark brown colored mixture was formed. The mixture in a crucible was put a microwave oven with an irradiation power of 800 Watt. In the microwave-assisted combustion, the precursor solution with fuel boiled and starting released a lot of gases of which  $CO_2$ ,  $NO_2$ , and  $N_2$  after dehydration accompanied by simultaneous decomposition. As the solution reached the point of spontaneous combustion, it started to burn and release heat, causing the solution to vaporize and turn into solid material. Finally, filter papers were used so that compound filtered. Powder that Product was washed via ethanol and distilled water for (3) times, dried at (70 °C) for 2 hours. Finely mortar was used to grind the resulting powder to obtain fine powders.

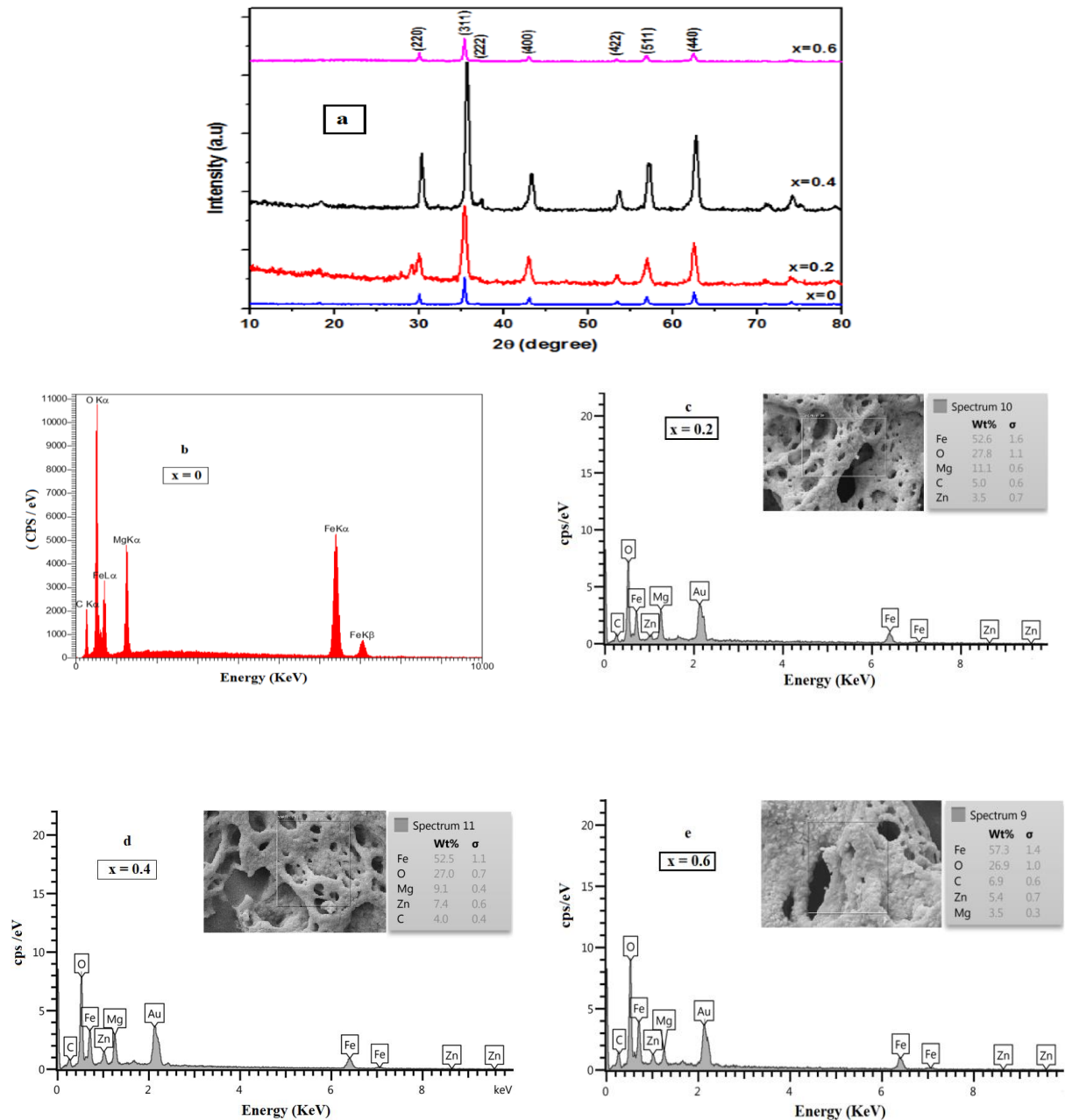
The X-ray diffraction analyses were obtained from the (Shimadzu XRD 6000, Japan). Morphology and elemental analyses were examined with field emission- scanning electron microscope [TE-SCAN Mira3, France] for samples of  $x=0$ , and FE-SEM device kind [FEI Nova Nano SEM 450] for samples of  $x = 0.2, 0.4, \text{ and } 0.6$ . Finally, magnetic hysteresis for all ferrite samples were obtained with a vibrating sample magnetometer (VSM, MDK).

## 3. Results and Discussion

### 3.1 Structure and morphology characterization

$Mg_{1-x}Zn_xFe_2O_4$  ferrite series samples [where  $x = (0, 0.2, 0.4 \text{ and } 0.6)$ ] which were synthesized by microwave-assisted combustion route, were analyzed ( for structure and elemental analysis) using X-ray diffraction techniques (XRD) and energy dispersion X-ray analysis as shown in Figure (1. a, b, c, d, and e ). The peak positions of  $Mg_{1-x}Zn_xFe_2O_4$  in

XRD patterns [which are shown in Figure (1. a)] are indexed to (220), (311), (222), (400), (422), (511) and (440) which corresponds to the spinel phase of ferrite structure. Similar findings have been in other studies[12,13]. Our results of the XRD patterns were in agreement with the standard data (JCPDS PDF card No. 01-077-0011). From XRD pattern, it can be observed that the sample with  $x=0.4$  crystallizes more than the other samples. This is may be due to the synthesis conditions such as reaction time in the microwave oven, or slower ignition of the exothermic reaction using microwave energy, which prevent the slower simultaneous decomposition of the metal nitrate, which enhances the formation of phase.



**Figure 1-**XRD and EDX pattern for mixed spinel  $Mg_{1-x}Zn_xFe_2O_4$  ferrite.

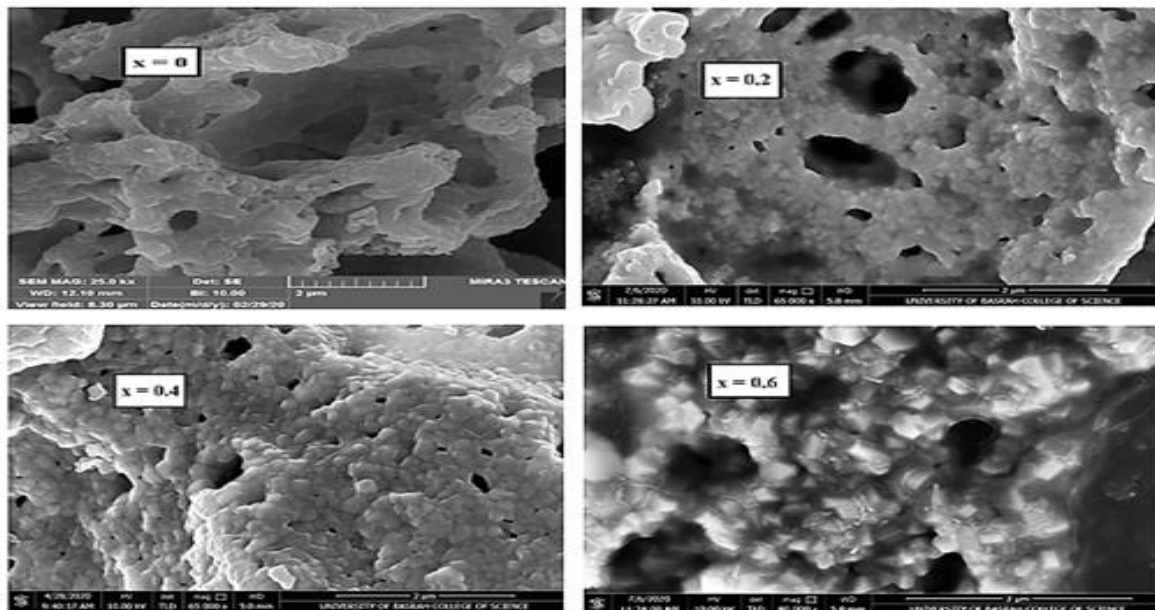
The crystallite size and lattice constant were calculated from XRD line broadening of the highest peak (311) plane using Deby-Scherrer formula [14] is given in Table 1.

**Table 1-** Crystallite size and lattice constant of the  $Mg_{1-x}Zn_xFe_2O_4$  ferrite series samples.

As-synthesized Powdered Samples	x	Lattice constant a (Å)	Crystallite Size (XRD) D(nm)
$MgFe_2O_4$	0	8.4003	30.66
$Mg_{0.8}Zn_{0.2}Fe_2O_4$	0.2	8.4320	16.23
$Mg_{0.6}Zn_{0.4}Fe_2O_4$	0.4	8.3564	33.65
$Mg_{0.4}Zn_{0.6}Fe_2O_4$	0.6	8.4026	26.53

From Table 1, it is clear that the lattice constant increases from 8.3564 Å to 8.4026 Å with the increase of  $Zn^{2+}$  ion concentration for series mixed spinel  $Mg_{1-x}Zn_xFe_2O_4$  ferrite except  $x = 0.2$ . which can be illustrated as an ionic radius of divalent magnesium ions  $Mg^{2+}$  (0.67Å) is smaller compared to divalent zinc ions  $Zn^{2+}$  ion (0.74Å) [12,15]. Similar observations were reported by Reyes-Rodríguez et al for Mg-Zn ferrite [16] and Mirshekari et al. for other spinel ferrites [17]. The elemental analyses for as-synthesized powdered samples of the mixed spinel are shown in Figure (1. b, c, d, and e). Fe, Mg, and O elements can be observed in Figure (1. b), while Figure (1, c, d, and e) showed the peaks because of Zinc (Zn) atoms in mixed spinel ferrite ( $x = 0.2, 0.4$  and  $0.6$ ) samples.

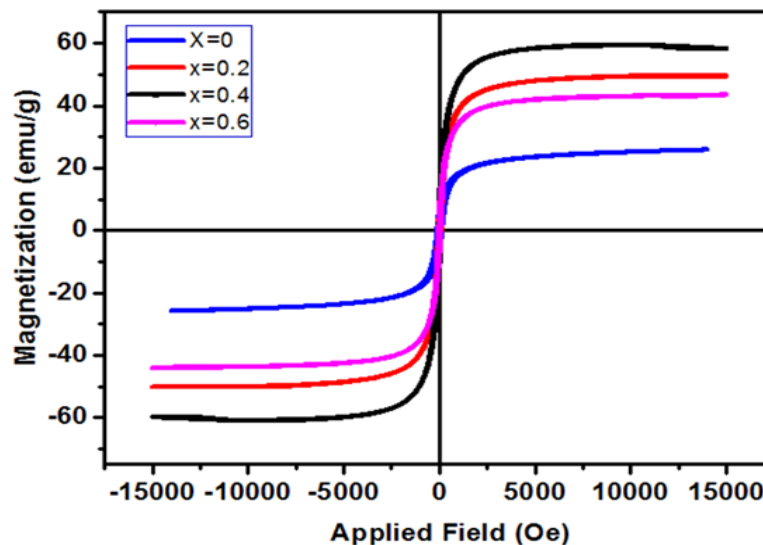
The morphology of mixed spinel  $Mg_{1-x}Zn_xFe_2O_4$  ferrite shows a uniform distribution of the nanoparticles, this may be because the samples are different in individual phases growth rate. FE-SEM images also showed presence porosities in the samples which may be because of release of gases during the combustion process. By comparing the results of the present work with that Rahmar[18], it was found that as-synthesized samples by this route almost displayed agglomeration.

**Figure 2-**FE-SEM images for powdered samples of the mixed spinel ferrite.

### 3.2 Magnetic Measurements

Information about saturation magnetization of the samples of the mixed spinel  $Mg_{1-x}Zn_xFe_2O_4$  ferrite where  $x = (0, 0.2, 0.4, \text{ and } 0.6)$  was determined by using a vibrating sample magnetometer with an applied external field of  $(15 \times 10^3 \text{ gauss})$  at room temperature, as shown in Figure 3. The magnetization curve showed the lowest saturation magnetization (25.97 emu/g) of the ferrite sample for  $x = 0$ . This low net magnetization value is mostly due to  $(Mg^{+2})$  moments at B- site only. thus, moments of  $Fe^{+3}$  from the A- sites and B- sites cancel each other, this behavior causes low magnetization value. Also, the magnetization curve in

Figure 3 of the ferrite sample for  $x = 0.2, 0.4,$  and  $0.6$  shows increase in saturation magnetization as zinc ions ( $Zn^{+2}$ ) concentrations increases. As magnesium ions ( $Mg^{+2}$ ) are replaced by zinc ions ( $Zn^{+2}$ ), saturation magnetization changes. It is observed that the value of saturation magnetization increased as the substituting with zinc content increases from  $49.51\text{emu/g}$  to  $59.55\text{ emu/g}$  at as  $Zn^{+2}$  concentrations increase from  $x = 0.2$  to  $x = 0.4$ . Saturation magnetization shows a decreasing trend for mixed spinel ferrite at  $x = 0.6$  due to the  $ZnO$  secondary phase, which was also observed by other authors [19,20]. The explaining of this phenomenon is that  $Mg_{0.4}Zn_{0.6}Fe_2O_4$  has an inverted spinel crystal structure. The observed changes in saturation values with increasing the zinc content is illustrated as the specific basis for exchange interactions that occur between various ions at octahedral and tetrahedral sites in spinel ferrites, Similar results were obtained by Mirshekar et al[14] and Yahya et al[21]. Net magnetization arises from the variation between the magnetic moments at octahedral and tetrahedral sites. In nature, both divalent magnesium ions ( $Mg^{+2}$ ) and divalent zinc ions ( $Zn^{+2}$ ) are non-magnetic, where  $Zn^{+2}$  ions located in tetrahedral A site, while  $Mg^{+2}$  ion is located in the octahedral B site. When divalent Zinc cation is replaced by divalent magnesium cation, it forces  $Fe^{+3}$  ion at the octahedral site to migrate to the tetrahedral site which results in a decrease of the net magnetic moment, thus saturation magnetization ( $M_s$ ) decreases with the decrease of magnesium content (increase zinc content). Our results are similar to those of Rahman et al. [19]. Generally, the smaller value of saturation magnetization is usually attributed to lattice defects, surface spin disorder, and the presence of a magnetically inert layer on the nanoparticles' surface. Iron ions in the superficial layer have a random orientation of their magnetic moments due to their broken bonds, resulting in a decrease in the overall magnetization of each individual nanoparticle [22]. Saturation magnetization of the mixed spinel  $Mg_{1-x}Zn_xFe_2O_4$  ferrite are given in Table 3.



**Figure 3-**Magnetic hysteresis curve of the mixed spinel  $Mg_{1-x}Zn_xFe_2O_4$  ferrite samples.

**Table 2-** Saturation magnetization of the mixed spinel  $Mg_{1-x}Zn_xFe_2O_4$  ferrite where  $x = (0, 0.2, 0.4,$  and  $0.6)$

as- synthesized powdered samples	$M_s$ (emu/g)
$MgFe_2O_4$	25.97
$Mg_{0.8}Zn_{0.2}Fe_2O_4$	49.51
$Mg_{0.6}Zn_{0.4}Fe_2O_4$	59.55
$Mg_{0.4}Zn_{0.6}Fe_2O_4$	43.58

#### 4. Conclusion

In the present work, mixed spinel  $Mg_{1-x}Zn_xFe_2O_4$  ferrite, where  $x = (0, 0.2, 0.4, \text{ and } 0.6)$ , was synthesized using the microwave - assisted combustion route. It was found that: 1) Microwave-assisted combustion route that is usually performed in solution media has short time reaction and simplicity. 2) Lattice parameter increased linearly with increasing the Zn content, 3) the  $Mg_{1-x}Zn_xFe_2O_4$  ferrite showed S-shape hysteresis curve, 4) Pure magnesium ferrite has low net magnetization value but when magnesium ions ( $Mg^{+2}$ ) were replaced by zinc ions ( $Zn^{+2}$ ), an increase in saturation magnetization for mixed spinel ferrite at  $x = 0.2$  and  $0.4$  was observed and then a decrease in saturation magnetization for inverted spinel ferrite at  $x = 0.6$ .

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