Republic of Iraq

Ministry of Higher Education

and Scientific Research

University of Diyala

College of Science

Department of Physics

Structural, Electrical and magnetic properties of superparamagnetic TiO_2 mixed $Zn_xMn_{1-x}Fe_2O_4$ Nanoferrites

A Thesis

Submitted to The Council of the College of Sciences, University of Diyala in Partial Fulfillment of the Requirements for the Degree of Master of science in Physics

By

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2022 AD ***1443AH***

بِسْمِ ٱللَّهِ ٱلرَّحْمَٰنِ ٱلرَّحِيم

(أَوْ كَظُلُمَٰتٍۢ فِى بَحْرٍۢ لُّجِّىٍّۢ يَغْشَىٰهُ مَوْجٌۭ مِّن فَوْقِهِۦ مَوْجٌۭ مِّن فَوْقِهِۦ سَحَابٌۭ ۚ ظُلُمَٰتٌۢ بَعْضُهَا فَوْقَ بَعْضٍ إِذَآ أَخْرَجَ يَدَهُۥ لَمْ يَكَدْ يَرَىٰهَا ۗ وَمَن لَّمْ يَجْعَلِ ٱللَّهُ لَهُۥ نُورًۭا فَمَا لَهُۥ مِن نُّورٍ ۝)

صدق الله العظيم

سورة النور

الآية 40

Dedication

My M.Sc. is dedicated to…

My merciful parents.

My lovely wife.

My beautiful children.....Ellen and Taleen

Muthana El ttayef Abbas

2022

Acknowledgments

I would like to express my sincere gratitude to the Assistant supervisor ***Assistant Professor Zena Mohammed Ali abbas***, thank ***Prof. Dr. Tahseen Hussein Mubarak***,***Dr.Ziad Tariq Khodair*** thank ***Dr.Sabah Anwer Salman, Dr. AmmarA. Habeeb*** thank ***Dr.Mohammad Hameed*** and all my professors in the Physics department who gave me the opportunity. perform this research. I am indebted to them for their suggestions and valuable remarks.special thanks are extended to the Dean of the College of Science and all the staff of the Department of Physics for their assistance.

To the one who honored me by bearing his name, ***my Dear father***.. To the light of my eyes, the light of my path, and the joy of my life**, *my mother***, To ***my dear wife***, thank you for always being by my side, taking care of me and supporting me from the bottom of my heart. Thank you for every moment that you accompanied me during my study period and for your encouragement to me. I ask God to give you goodness and open the doors of His mercy for you. To my beautiful flowers, ***Elaine*** and ***Taleen***.and to my supporters ***brothers*** and ***sisters***.I certainly cannot mention the names of everyone who contributed towards the success of this research, but as they have always known, I am very grateful.

Muthana El ttayef Abbas

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Supervisors Certification

I certify that this thesis entitled, ***Structural, Electrical and magnetic properties of superparamagnetic TiO_2 mixed $Zn_xMn_{1-x}Fe_2O_4$ Nanoferrites***, for the student (***Muthana Elttayef Abbas***), was prepared under supervisions at the Department of Physics, College of Science, Diyala University in part, of the prerequisites for the awarding the M.Sc in Physics.

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We certify that we have read this thesis entitled " ***Structural Electrical and magnetic properties of superparamagnetic titanium doped Mn-Zn nanoferrites for high frequency applications*** " and, as an examining committee, we examined the student (***Muthana El ttayef Abbas***) on its content, and in what is related to it, and that in our opinion it meets the standard of a thesis for the degree of master in Physics Sciences.

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Symbol	***Definition***
θ	Bragg's angle
λ	Wavelength
M	Magnetization
H	Magnetic field
M_S	Saturation magnetization
H_c	Coercivity
M_r	Remanence magnetization
n_B	Magnetic moment
K	Magnetic anisotropy
T	Temperature
a	Lattice parameters
ε'	Dielectric constant
ε''	Dielectric loss factor
$tan\ \delta$	Dielectric loss angle
σ_{ac}	ac conductivity
T_c	Curie temperature
T_N	Neel temperature,
μ_r	Relative permeability
μ_o	Vacuum permeability

μ	Permeability of specific medium
χ	Susceptibility
χ_m	Magnetic susceptibility
c	Curie constant.
P	Polarization
C	Capacitance
C_o	Capacitance of air
f	Frequency
d	Spacing between the atomic planes
D	Crystallite size
hkl	Miller indices
ρ_x	X-ray density
M	Molecular weight
℃	Degrees celsius
P_e	Electric polarization
P_i	Ionic polarization
P_o	Orientation polarization
P_S	Space-charge polarization
D_c	Critical diameter
α	Polarizability
E_a	Activation energy

List of Abbreviations

Abbreviations	***Definition***
NPs	Nanoparticles
NCs	Nano composites
XRD	X-ray diffraction
TEM	Transmission electron microscopy
FT-IR	Fourier transform infrared spectroscopy
SEM	Scanning electron microscopy
FE-SEM	Field emission-scanning electron microscopes
VSM	Vibrating sample magnetometer
MRI	Magnetic resonance imaging
FWHM	Full width at half-maximum
PXRD	Powder X-ray diffraction
GMR	Giant magneto resistance

Abstract

The objective of this thesis is to synthesize and study the structural, magnetic and electrical properties of nanoparticles made of ferrite zinc manganese using the stoichiometric formula $Zn_xMn_{1-x}Fe_2O_4$ (X= 0, 0.15, 0.25, 0.35, and 0.45) respectively, were prepared using the sol-gel auto-combustion process. After combustion, the as-burnt powders were calcined at 650, 750, and 850 °C for 2 hrs to increase homogeneity and remove organic waste, where the as-burnt specimens and the specimens that calcined at 650, 750, and 850 ˚C added with a five-drop PVA as a binder to press it into circular pellets of diameter 1cm with thickness about 3 mm. The prepared pellets were sintered at 400, 600, and 800 ℃ for 2 hrs to intensify of the specimens and, slowly allowed to be cooled naturally to examine the dielectric properties. Another purpose of this work is to blend and study the structural, magnetic and electrical properties of zinc manganese nitrate ferrite and titanium nanocomposites at 500 using the formula $ZnMnFe_2O_4/TiO_2$ in different proportions (20%, 30%, 40%, 50% and 60%) respectively. prepared using the traditional ceramic method. The best results were the crystal size in this paper with a size of 15 to 38 nm with a cubic structure which was determined by XRD, while the crystal size of Mn-Zn-ferrites/TiO2 was from 10 to 30 nm with a cubic structure determined by X-ray diffraction (XRD) analyses. FE-SEM reveled Nano ferrites ($ZnMnFe_2O_4$) having a particle size of 25 to

66 nm were found. While the particle size of ZnxMn1-xFe_2O_4 /TiO_2 the range from 37 to 72 nm. It observed the result is less than the Zn-Mn-Fe_2O_4 nano composite.

Prominent FT- IR absorption peaks at 400 and 500 cm^{-1} indicated the presence of Fe-O, Mn-O, and Zn-O vibrational bands. Results from the VSM demonstrated that the magnetic nature of materials changed dramatically from pristine as magnetization rose and coercively decreased with Mn-Zn-ferrites nanoparticles. Finally, an optimal magnetic parameter value (Ms =0.80 emu/g and Hc =75 Oe) at (850) °C was obtained from the data. The dielectric properties are measured using a (LCR) meter in the frequency range of (200Hz-1MHz) at room temperature. The dielectric constant (ε'), dielectric loss angle (tanδ) and dielectric loss factor (ε'') are for Mn-Zn-Fe_2O_4 nanoferrites at 400, 600, and 800 ℃ and Mn-Zn-Fe_2O_4/TiO_2 nanocomposites in different proportions (20%, 30%, 40%, 50% and 60%) respectively are found to decrease with increasing temperatures. The prepared pellets were sintered at 500 ℃ for 2 hrs to intensify of the specimens and slowly allowed to be cooled naturally to examine the dielectric properties.

Chapter one

Introduction & Literature Review

1.1 Introduction

Researchers are continuous work on new materials that could be applied to a range of industries. fabric, glass, wood, metals, ceramics, alloys, petroleum fuels, radioactive materials, polymers, coal, stone, semiconductors, and other materials have ushered in significant advances in humanity's history. The investigation of innovative materials with superior properties, on the other hand, stretches back to the stone age. The study of material synthesis and properties has only recently emerged as a distinct field of science with technical and practical ramifications. In the domains of physics and other sciences, nanotechnology is one of the most important and intriguing technologies. It has made a significant contribution to the events of great scientific revolutions that are hoped to change the course of technologies and applied sciences, as it provides a high ability to settings and control in the composition of matter at the level of atomic dimensions, as well as a high potential in nanofabrication, resulting in amazing physical qualities and properties. because of this nanotechnology has been used to create systems and devices with unique features by manipulating the form and size of the nanosphere [1]. Due to the enormous ratio of the surface of the grains to their size, magnetic materials in general, and nanoferrite in particular, have a significant impact on physical, electrical, and magnetic properties. Due to its magnetic properties and a wide range of uses, nanoferrite has sparked interest in the sphere of science and technology in recent years [2]. High frequency transformer cores, antenna bars, and choke coils are all made of ferrites [3,4]. nanoelectronic devices, integrated circuits, and magnetic resonance imaging (MRI) are all examples of this [5-8]. The typical formula for ferrites is (MFe_2O_4), where M represents , one of the divalent metallic elements (Zn^{+2},Cu^{+2},Fe^{+2},Mg^{+2}). The ferrites are divided into three groups based on their chemical composition: Garnet, Hexagonal and Spinal ferrite [9]. Because of its strong electrical and magnetic properties and wide range of uses, we will concentrate our research on this last type. Spinal ferrites are materials with good magnetic and electrical properties that are highly influenced by the distribution pattern of positive ions (cations) between the tetrahedral and octahedral sites [10]. One of the most important methods of preparing nanoferrite is the sol-gel method auto-combustion since it is simple to prepare, takes little time, and does not require high temperatures [11]. Ferrite is made from a powder that is compressed and sintered to take the desired shape. It is one of the simplest and cheapest materials to make, and its properties are determined by a number of factors, including the shape and size of the grains, the method of preparation, the sintering temperature, the type of materials that make up ferrites, and their quantity [12]. In this study, the synthesis of Zn-Mn-Fe_2O_4, and Zn_xMn1-xFe_2O_4 /TiO_2 nanocomposite by Sol-Gel method at different temperatures (650, 750, and 850) °C Nano-size Mn–Zn particles were synthesized by the sol–gel combustion method. These particles were prepared from metal nitrates and citric acid. Also, the Zn-Mn-Fe_2O_4, and Zn_xMn1-xFe_2O_4/TiO_2 nanocomposite were characterized by (XRD) (XRD-6000/Japan) and the results compare with the JCPDS card (Joint Committee on Powder Diffraction Standards), (FE-SEM) by "(Tescan Mira3 SEM-Czechia)" in Iran-Mashhad, (VSM), (LCR) for study structure, magnetic, and electrical properties.

1.2 Literature Review

The study of nanospinel ferrite has piqued the interest of researchers in recent years due to its unique properties and wide variety of applications. The following is a list of some works on the properties of nanospinel ferrites that have been stndied, including structural, electrical, and magnetic properties:

In 2010 Bhalla et al., manufactured Mn-Zn soft ferrites by powder metallurgy and overall output yield of it’s plant. The efforts have been made to synthesize the crucial parameters which are responsible for better material preparation, pressing and sintering. By adopting these recommendations, the rejection rate is substantially reduced and the variation in magnetic properties is less. Data which give more uniformity in bigger lots and are responsible for more uniform magnetic properties, have been discussed. Simple, quality-control instruments and their measurement methods which can be incorporated for stage inspection have been explained. The additives for better ferrite powder preparation, granules making and to obtain better magnetic have been discussed. Improved pressing, sintering, porosity, density and permeability relationship have been drawn. A sintering method to obtain better sintered density and high permeability in ferrites is also explained [13].

In 2012 Deraz and Aarifi, prepared Co_{1-x} $Zn_xFe_2O_4$ used sol-gel auto combustion to make nanocrystalline Zn-substituted cobalt ferrite powders, (x = 0, 0.25, 0.5, 0.75, and 1). The specimens had a cubic spinel structure, and the X-ray diffraction investigation revealed that the crystallite size decreased from 70 to 51nm as the zinc content was increased to (x=1).The lattice constant increased from (0.8370 to 0.8400 nm) with increasing the concentration of zinc to (x=1), while the X-ray density increased from (5.293 to 5.381 g/cm^3) as the concentration of zinc increased to (x=1). The saturation magnetization of Co-Zn nanoferrites was examined using a vibrating sample magnetometer (VSM) at room temperature, and the results showed that the saturation magnetization increased as zinc substitution increased. Increased Zn concentrations resulted in a drop in coercivity (Hc) from 807.7 to 46.0 Oe [14].

In 2011 Varshney et al.,The prepared $Zn_xMn_{1-x}Fe_2O_4$ (x = 0.0, 0.25, 0.50, 0.75, 1.0) mixed ferrites, by chemical co-precipitation method. and studied crystal structure, average crystallite size, Raman spectra and magnetic properties, The X-ray diffraction pattern confirms that the mixed ferrite samples are in cubic inverse spinel structure. The variation of lattice parameter with increased Zn doping concentration illustrates a decreasing trend. The mixed ferrite crystallite size gradually increases with enhanced Zn doping concentration. Porosity shows decreasing trend with increased Zn doping concentration and confirms that the synthesized samples have dense random packing. The absorption band at about 500–600 cm^{-1} in the IR spectra corresponds to the vibration of Fe^{2+}–O^{2-} bond related to tetrahedral (A) site without any traces of impurity (NO_3) peak. [15].

In 2012 Arana et al., The Studied structural and magnetic properties after different thermal treatments, Spinel ferrites of composition $Zn_{0.6}Mn_{0.4}Fe_2O_4$ and $Li_{0.2}Zn_{0.2}Mn_{0.4}Fe_{2.5}O_4$ were prepared by the self-combustion sol-gel method. The samples were heat-treated in different atmospheres and temperatures, producing different effects on their morphological and structural properties. The resulting products of each treatment were structurally and magnetically characterized. Incorporating Li to the crystalline lattice increases saturation magnetization and promotes a decrease in secondary phases segregation. This result is explained assuming that Li incorporation produces a cationic redistribution in the spinel structure [16].

In 2013 M. Zhang et al., The Studied and prepared $Ni_{0.5}Zn_{0.5}Fe_2O_4$ by the sol-gel method to (9) specimens of the chemical. After completing an X-ray diffraction investigation, it was discovered that all specimens of the produced ferrite compound formed the spinel phase, with average crystallite sizes ranging from 9 to 96 nm, if the average crystallite size increasing with the increase in the annealing temperature. When the annealing temperature is raised, the lattice constant decreases. The results of the magnetic measurements performed on the specimens of the synthesized chemical revealed that all of the specimens were paramagnetic. It was also discovered that when particle size rises, saturation magnetization increases, which can be explained by action redistribution on tetrahedral A and octahedral B sites, as well as domain wall motion [17].

In 2014 Veverka et al., The prepared $Co_{0.4}Zn_{0.6}Fe_2O_4$ by the co-precipitation method to make magnetic cores of two different sizes, which were annealed at temperatures of 500 °C and 650 °C. The nanoparticles were encapsulated in silica, which resulted in colloid ally stable water suspensions. The increase of annealing temperatures had caused a significant rise in Curie temperatures (Tc) and blocking temperature (T_B), additionally the heating efficiency of sample had been enhanced [18].

In 2014 Rajendra et al., The studied the structural and electric properties of Titanium Substituted Ni-Cu-Zn Ferrite by solid state reaction. In work, the impact of Ti^{4+} substituted $Ti_x(Ni_{0.35}Cu_{0.05}Zn_{0.60}Fe_{1.98}O_{4+\delta})_{1-x}$ ferrite is investigated by varying the concentration of the Ti^{4+} with x = 0.003, 0.0765, 0.15. The compound is synthesized by solid state method. Structural and electrical properties have been explored using XRD, FTIR, and dielectric spectroscopy technique. X-ray diffraction (XRD) patterns reveal the formation of the cubic spinel phase in the samples after sintering the compound at 10000C The mean crystallite size DXRD of the samples determined from XRD line broadening is 35.18–44.68 nm. The dielectric constant vary as a function of frequency and composition at room temperature. It is observed that with the increase in the concentration of Ti4+ ions dielectric constant increases [19].

In 2015 Joseyphus et al., The studied and prepared $Mn_{0.67}Zn_{0.33}Fe_2O_4$ nanoparticles with size ranging from 20 to 80 nm using the modified oxidation method. The Curie temperatures for all the samples are found to be within 630 ± 5 K suggesting that there is no size-dependent cation distribution. The critical particle size limit for super paramagnetism is found to be 25 nm at 293 K. The modified oxidation method can be used to synthesise ferrite nanoparticles of the required size range with specific magnetic properties suitable for applications [20].

In 2016, C. Stergiou, The studied microstructure and electromagnetic properties of Ni-Zn-Co Ferrite up to 20 GHz, The present paper examines the relation between different developed microstructures and the microwave electromagnetic properties in Ni-Zn-Co ferrite. To this end, the $Ni_{0.25} Zn_{0.25} Co_{0.5} Fe_2 O_4$ composition has been prepared with the conventional ceramic process with varied prefiring T_P (750∘C, 1000∘C) and sintering T_S top temperatures (1200 ∘C, 1250 ∘C). When lower temperatures are applied in these production stages, incomplete microstructures with low density, higher porosity, or finer grains are achieved. On account of these features, the contributions of domain wall motion and spin rotation to the complex permeability μ^* move to higher frequencies, whereas microwave dielectric permittivity ε^* is decreased. In particular in conjunction with the high Co content, the wall relaxation and spin resonance are interestingly forced to occur at 850 MHz and 8.05 GHz, respectively. [21].

In 2016 Singh et al., The studied and prepared $Mg_xZn_{1-x}Fe_2O_4$ (x=0.5, 0.6, 0.7) by co-precipitation technique and then subsequently heated to 800 °C in order to investigate structural, thermal and magnetic properties. The samples are characterized by using XRD, FTIR, TGA-DSC, SQUID and Mössbauer spectroscopy techniques. The synergic effect of heat treatment with substitution of Mg^{2+}, results in random variation of lattice parameter (a) and crystallite size (D). FTIR studies revealed the formation of cubic spinel structure. The broadening at octahedral bands for compositions x=0.6 and 0.7 attributes to distribution of ferrite particles of different sizes in these samples. The characteristic feature of hysteresis loops reflects the nature of ferrite particles in the state of super para magnetism. The saturation magnetization at room temperature has been reported for composition x=0.7 is 44.03 emu/g. [22].

In 2017 Angadi et al., The studied and prepared $Mn_{0.5}Zn_{0.5}Sc_yFe_{2-y}O_4$ (y=0.00, 0.01, 0.03 and 0.05) nanoparticles by solution combustion method using mixture of fuels were reported for the first time. The mixture of fuels plays an important role in obtaining nano crystalline, single phase present without any heat treatment. X-ray diffraction (XRD) results confirm the formation of the single-phase ferrites which crystallize in cubic spinel structure. The Fourier transform infrared spectra

(FTIR) exhibit two prominent bands around 360 cm^{-1} and 540 cm^{-1} which are characteristic feature of spinel ferrite. The transmission electron microscope (TEM) micrographs revealed the nanoparticles to be nearly spherical in shape and of fairly uniform size. The room temperature impedance spectra (IS) and vibrating sample magnetometry (VSM) measurements were carried out in order to study the effect of doping (Sc^{3+}) on the characteristic properties of Mn-Zn ferrites. Further, the frequency dependent dielectric constant and dielectric loss were found to decrease with increasing multiple Sc^{3+} concentration. Magnetic measurements reveals that saturation magnetization (M_s), remnant magnetization (M_r), magnetic moment (η_B) and magnetic particle size (D_m) increase with Sc^{3+} ion concentration up to x=0.03 and then decrease. The values of spin canting angle (α_{Y-K}) and the magnetic particle size (D_m) are found to be in the range of 68–75° and 10–19 nm respectively with Sc^{3+} concentration. [23].

In 2019 Petrescu et. al, The studied the morphological, structural, and chemical composition of the $Mn_XZn_{(1-X)}Fe_2O_4$ ferrites are produced by using a mixture of finely-powdered nitrogen-based salt precursors pressed into a mold, and subsequently sintering the resulting powder. The bulk samples are cut into smaller prismatic shapes (rectangular prisms) of various volumes, by using a diamond coated steel wire. The magnetic characterization of the MnZn type ferrite provides one with valuable information, as the open sample results have been corrected with demagnetization factors, which consider the punctual susceptibility of the material. The novelty of this work consists of the estimation of these factors by an iterative procedure, i.e., by using an exponential fitting procedure. The corrected values are presented and discussed and fit manufacturers' data, which are provided as a range of values. The last part of the study focuses on estimating the magnetic losses in high frequencies for this ferrite by using the Steinmetz approach, i.e., the hysteresis losses are determined for the MnZn ferrite. All these properties make MnZn-type ferrites ideal for applications in a variety of high frequency transformers, adjustable inductors, wide band transformers, and high frequency circuits from 10 kHz to 50 MHz [24].

In 2020 Vahdati and Sedghi, studied the properties of zinc-manganese nanoferrite to compare organic fuels that were produced in conditions created by the auto gel combustion method, using citric acid, glycine, and urea with different pH. The samples were prepared in stoichiometric ratios to gain $Mn_{0.5}Zn_{0.5}Fe_2O_4$, and all the samples were calcined in the same condition (500 °C and 30 minutes). It should be noted that the entire process of synthesis was photographed to analyze the effect of fuels during the combustion process. Combustion reactions were studied by simultaneous thermal analysis (STA), FT-IR spectroscopy, and X-ray diffraction (XRD), also the rietveld method was used to determine the type and amount of crystalline phases. Magnetic properties of the samples were measured by vibration sample magnetometer (VSM), and their morphology and powder agglomeration was observed by field emission scanning electron microscopy (FE-SEM) and transmission electron microscopy (TEM). Superior magnetic properties of the sample synthesized with glycine were achieved. Urea gave the smallest particle size, while citric acid produced intermediate properties [25].

In 2021 Jahan et al., In the studyd, Al^{+3} substituted Ni−Zn−Co nanocompositions were successfully synthesized through the sol−gel technique. Rietveld refinement X-ray diffraction data evidenced the formation of the nanospinel structure with phase group Fd3m for all of the compositions. The cation distributions were established through the Rietveld refinement technique. Average particle sizes were in the regime of 25−29 nm, as demonstrated by TEM examinations. Thin areas of Al−O−Al structures were formed around the grain boundaries that hindered particle growth. The vibrational modes' characteristic peaks are red-shifted and blue-shifted, as exhibited in the samples' Raman spectra. Enhanced Ms with low values of Hc and Mr was found for the compositions. Magnetic outcomes revealed that the prepared nanocompositions are soft ferromagnetic materials and suitable for numerous technological applications. Frequency-dependent dielectric constants and ac resistivity indicated that the ferrites were highly resistive. Introducing Al^{3+} in Ni−Zn−Co ferrites makes the nanosamples highly resistive while maintaining the high magnetizations 86.39 emu/g for the x = 0.12 compositions. [26].

In 2021 Akhter et. al, prepared and studied $Cu_{1-x}Zn_xFe_2O_4$ (where x = 0.0–1.0 with step of 0.1) ferrites by double sintering solid state reaction method to explore disorder magnetic behavior. The room temperature (300 K) and low temperature (5 K) magnetic behavior has been analyzed using M-H curve, B–H loop, μi-T curve and M-T curve. The saturation magnetization for sample x = 0.0 to 0.6 and non-saturation magnetization for sample x = 0.7 to 1.0 designate the existence of long-range and short-range ferromagnetic order respectively. The Curie temperature has been derived from the sharp fall of permeability for sample x = 0.0–0.6 which is declining with raising Zn content due to redistribution of cations and weakening of A-B exchange interaction. The shrinking of coercivity affirms softer nature which can be used as high frequency soft magnetic materials. For M-T curve, when temperature is lowered from 300 K to 5 K, different disorder magnetic transition such as paramagnetic- ferromagnetic-reentrant spin glass-spin glass has been remarked for sample x > 0.6. All these feature of mag- netic behavior of Cu–Zn ferrites has been studied in details in this paper [27].

1.3 Aim of the Study

1- Synthesis of $Zn_xMn_{1-X}Fe_2O_4$ nanoferrites by a sol-gel auto-combustion method and $Zn_xMn_{1-X}Fe_2O_4$ /TiO_2 by the conventional ceramic method.

2- Studying the effect of increasing the content (x) and calcination, temperature of $Zn_xMn_{1-X}Fe_2O_4$, and studying the influence of (TiO_2) concentration on the structural, magnetic, and dielectric properties (XRD, FT-IR, FE-SEM and EDS).

3- Testing the magnetic parameters such as (saturation magnetization (Ms), remanence magnetization (Mr), and coercivity (Hc)) of synthesized ferrite nanoparticles in applied field ±15 kOe through vibrating sample magnetometer (VSM) at room temperature.

4- Measurments of the electrical properties (dielectric constant (ε'), dielectric loss angle ($tan\ \delta$), dielectric loss factor (ε''), and ac conductivity (σac)) of synthesized nanoferrite with the frequency from (50 Hz) to (2MHz) by using LCR meter at room temperature.

5- Magnetically flexible ferrites are used in cores for transformer windings, electric motors, and transceiver antennas, In HF technology, in the construction of antennas and in parts for moderators, In headphones for electronic recording equipment, video equipment, and hard disks,and Stealth and camouflage technology (ferrites absorb radar waves).