Republic of Iraq Ministry of Higher Education & Scientific Research University of Diyala College of Sciences Department of Physics



# Preparation of nano barium ferrite and study of its physical properties

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 sciences in Physics

> By Nisreen Zaidan Khalaf B.Sc. in Physics (2010-2011)

# Supervised by

Assist. Prof. Dr. Tahseen H. Mubarak

Prof. Dr. Karim H. Hassan

**2014 AD** 

1435 AH

َقُولُ تعالى عَزَّ وَجَلَّ: ويسألونك عن الروح قل الروح من أمر ربي وما أوتيتم من العلم إلاً قَلِيلاً ﴿85

الاية {85}

سورة الإسراء

# **DEDICATION**

To the three pillars of my life: my God, my father and my mother. Without you, my life would fall apart. I might not know where the life's road will take me, but Walking with You:

My God, through this journey has given me Strength.

<u>My Father</u>, I would like to lift your forehead high in response your grace to me.

<u>My Mother</u>, if love was a human it shall be you, thanks for your faith in me.

<u>My brothers</u> the source of my happiness and tranquility

<u>My lonely sister</u> I thank my Lord to make you sister to me, not to be sister to another else.

Thanks for inspiring. We made it...

<u>Nisreen</u>

# ACKNOWLEDGEMENTS

First of all I thank his Almighty Allah, whose Grace enabled me to continue this work and overcome all difficulties and our prophet Muhammad (peace and blessings of Allah be upon him) who invite us to science and knowledge.

I would like to express my sincere gratitude to my supervisors Assist Prof. Dr. Tahseen Hussein Mubarak and Prof. Dr. Karim H. Hassan who granted me the opportunity to do this research. I am indebted to them for their suggestions and valuable remarks.

Special thank are extended to the Iraq, university of Diyala, College of Science and her Dean and all the Staff of the Department of Physics for their assistance.

I am also grateful to the physics Department at the College of Education Ibn al-Haytham for their kind help during the laboratory work and x-ray tests also special thank for Ministry of Sciences and Technology for their help during the electric and mechanic tests.

My greatest indebtedness goes to my Parents for their valuable advice, my Brothers and my Sister and her husband for their endless support.

Finally, my deepest gratitude is dedicated to my brothers (Mohammed, Salam and Abdullah), and my sister (Rana), They were with me every breath and my dearest friends Thora, Amaal and my Studying Partners Rudaina, Asmaa, Noor, Zainab, Haydar, Aws, Nawar and Ahmed.

#### ABSTRACT

This research includes the preparing of barium ferrite ( $BaFe_{12}O_{19}$ ) of nano particle sizes as a magnetic materials, and also the study of some electrical properties such as dielectric constant and dispersion factor (loss tangent), and X-ray examination of the resulting material. Two methods to prepare nano barium ferrite using co-precipitation route using aqueous and (ethanol/water) solutions of iron chloride FeCl<sub>3</sub>.6H<sub>2</sub>O and barium chloride BaCl<sub>2</sub>.2H<sub>2</sub>O with a Fe/Ba molar ratio of 15.

Co-precipitated powders were dried at  $80^{\circ}$ C for 12 h and calcined at various temperatures for 2 h. Nano-size particles of barium ferrite with mean particle size of almost 30 and 70 nm are observed in the SEM micrographs of the samples synthesized in aqueous and ethanol/water solution after calcifying at 1000°C for 2h, respectively. Generally, the results of the tests show an increase in dielectric constant with the increase in frequency, and the values of dielectric constant of the samples prepared using the water are higher than the values of dielectric constant of the samples prepared using the solution (ethanol \ distilled water) with very little difference, this is because water has several impurities and needs a certain temperature to get rid of it resulting to high dielectric constant. The ethanol solution is characterized as volatile so it does not affect dielectric constant of material.

Also, the results show a decrease for both samples in the dielectric constant with increasing temperature and increasing frequency. And it's observed that the values of loss tangent decrease with increasing in frequency. The X-ray tests have explained that there is a change in phases of each sample with increase temperature; and it has shown that the grains are hexagonal and irregular shapes it similar to results that other researchers. Bulk density measurements are performed using law of density and it show there is an increase in the density and shrinkage of the sintered pellets with an increase of times and temperatures. Resistivity of ferrites is known to depend upon the purity of the starting materials and the preparation details such as sintering temperature; the resistivity of the ferrites is expected to decrease with the increasing sintering temperature.

### CONTENTS

Subject	Page
Chapter One: Introduction & Previous Studies	
1.1 Introduction	1
1.2 Previous Studies	4
1.3 Aims of the Study	15
Chapter Two: Theoretical Background	
2.1 Introduction	16
2.2 Nanotechnology	16
2.3 Production of nanoparticles and nanomaterials	18
2.4 Properties of nanoparticles	19
2.5 Precipitation processes	19
2.6 Ferrite and ferrite's structure	20
2.7 Magnetic materials types	21
2.7.1 Soft magnetic materials	21
2.7.2 Hard magnetic materials	22
2.8 M-Type ferrites	24
2.9 Intrinsic magnetic properties of M-Type ferrites	25
2.10 Structural Properties of ferrites	25
2.10.1 Debye-Scherrer-Method	26
2.10.2 Spacing of Lattice Planes	26
2.11 Mechanical properties of ferrites	27
2.11.1 Densities of ferrites	27
2.12 Diameter of grains	28
2.13 Electrical properties of ferrites	28
2.13.1 Dielectric constant	28

2.13.2 Electrical resistivity	29
2.13.3 Dielectric loss	30
2.14 Applications of the hard ferrites	33
Chapter Three: Experimental Part	
3.1 Introduction	38
3.2 The materials used	38
3.3 Method of preparation of barium ferrite in aqueous solvent	38
3.4 Method of preparation of barium ferrite using ethanol/water	40
solvent	τυ
3.5 Equipment (mold)	42
3.6 Forming	42
3-7 Sintering	43
3.8 Instruments	
3.9 Electric Tests	
3.9.1 Dielectric Constant	46
3.9.2 Dispersion Factor (Tangent Loss)	46
3.9.3 Electrical resistivity	46
Chapter Four: Results & Discussion	
4.1 Introduction	47
4.2 X-ray Diffraction analysis	
4.2.1 XRD pattern of the barium ferrite prepared using aqueous	
solutions	47
4.2.2 XRD pattern of the barium ferrite prepared using	50
ethanol/water solution	
4.3 Scanning electron microscope (SEM) test	54
4.4. Electrical measurements	60
4.4.1 Measurements of electrical resistivity ( $\rho$ ) and electrical	60

4.4.2 Measurements dielectric constant ( $\mathcal{E}_r$ ) and electrical loss tangent (tan $\delta$ )	(
4.5 Density test	,
Chapter Five: Conclusions & Recommendations	
5.1 Conclusions	,
5.2 Recommendations	,
Accessories	
References	, ,

## LIST OF TABLES

No.	Title	Page
(2-1)	A comparison of the Hard Ferrites.	23
(2-2)	Primary and secondary properties of M-Type ferrites	25
(2-3)	Common application of BaM ferrites	34
(3-1)	The chemical materials and their properties	38
(4-1 a)	Strongest three peaks of ferrite prepared in aqueous solution and calcined (at room temperature)	49
(4-1b)	Strongest three peaks of ferrite prepared in aqueous solution and calcined (at 800 °C)	49
(4-1 c)	Strongest three peaks of ferrite prepared in aqueous solution and calcined (at 1000 °C)	50
(4-2 a)	Strongest three peaks of ferrite prepared in ethanol/water solution and calcined (at room temperature)	53
(4-2 b)	Strongest three peaks of ferrite prepared in ethanol/water solution and calcined (at 800 °C)	53
(4-2 c)	Strongest three peaks of ferrite prepared in ethanol/water solution and calcined (at 1000 °C)	53
(4-3 a)	The values of bulk density of barium ferrite prepared in aqueous solution	71
(4-3 b)	The values of bulk density of barium ferrite prepared in ethanol/water solution	71

## **LIST OF FIGURES**

No.	Title	Page
(2-1)	Methods of nanoparticle production.	18
(2-2)	Mechanical-physical nanoparticle production processes.	19
(2-3)	Chemo-physical processes in nanoparticle production.	20
(2-4)	Hysteresis loop for hard magnetic materials.	23
(2-5)	Compositional phase diagram for hexagonal ferrites.	24
(2-6)	Schematic diagram for the types of polarization.	31
(2-7)	Show simplified diagram of currents in a loss dielectric	35
(3-1)	Preparation steps of barium ferrite nano powders in aqueous solution.	39
(3-2)	Diagram shows preparation of barium ferrite in aqueous solution	40
(3-3)	Preparation steps of barium ferrite nano powders using ethanol/water solution	41
(3-4)	Preparation of barium ferrite Using 3:1 ethanol/water solution	41
(3-5)	Mechanism of powder consolidation	42
(3-6)	Transformation of powder to compact sample	43
(3-7)	Powder metal compact before and after	44
(3-8)	LCR meter used in electrical measurement	45
(4.1 a)	XRD pattern of barium ferrite prepared in aqueous solution at room temperature	47
(4.1 b)	XRD pattern of barium ferrite prepared in aqueous solution and calcined at 800 °C	48
(4-1 c)	XRD pattern of barium ferrite prepared in aqueous solution and calcined at 1000 °C	48
(4-2 a)	XRD pattern of the barium ferrite prepared in ethanol/water solution at room temperature	51

(4-2 b)	XRD pattern of the barium ferrite prepared in ethanol/water solution and calcined at 800 °C	51
(4-2 c)	XRD pattern of the barium ferrite prepared in ethanol/water solution and calcined at 1000 °C	52
(4-3 a)	SEM images of barium ferrite prepared in aqueous solution and calcined at 1000 $^\circ\mathrm{C}$	54
(4-3 b)	SEM images of barium ferrite prepared in aqueous solution and calcined at 1000 $^\circ\mathrm{C}$	55
(4-3 c)	SEM images of barium ferrite prepared in aqueous solution and calcined at 1000 $^\circ\mathrm{C}$	55
(4-3 d)	SEM images barium ferrite prepared in aqueous solution and calcined at 1000 $^\circ\mathrm{C}$	56
(4-3 e)	SEM images of barium ferrite prepared in aqueous solution and calcined at 1000 $^{\circ}$ C	56
(4-4 a)	SEM images of barium ferrite prepared in ethanol/water solution	57
	and calcined at 1000 °C	
(4-4 b)	SEM images of barium ferrite prepared in ethanol/water solution and calcined at 1000 °C	57
(4-4 c)	SEM images of barium ferrite prepared in ethanol/water solution and calcined at 1000 $^\circ\mathrm{C}$	58
(4-4 d)	SEM images of barium ferrite prepared in ethanol/water solution and calcined at 1000 $^\circ\mathrm{C}$	58
(4-4 e)	SEM images of barium ferrite prepared in ethanol/water solution and calcined at 1000 $^\circ\mathrm{C}$	59
(4-5)	Resistivity as a function of frequency of barium ferrite prepared in aqueous solution and calcined at 800 °C	61
(4-6)	Resistivity as a function of frequency of barium ferrite prepared in aqueous solution and calcined at 1000 $^\circ \rm C$	61
(4-7)	Resistivity as a function of frequency of barium ferrite prepared using ethanol/water solution as a solvent and calcined at 800 °C	62
(4-8)	Resistivity as a function of frequency of barium ferrite prepared in	62

	ethanol/water solution and calcined at 1000 °C	
(4-9)	Conductivity as a function of frequency of barium ferrite prepared in aqueous solution and calcined at 800 °C	63
(4-10)	Conductivity as a function of frequency of barium ferrite prepared in aqueous solution and calcined at 1000 $^{\circ}$ C	64
(4-11)	Conductivity as a function of frequency of barium ferrite prepared in ethanol/water solution and calcined at 800 °C	64
(4-12)	Conductivity as a function of frequency of barium ferrite prepared in ethanol/water solution and calcined at 1000 °C	65
(4-13)	Dielectric constant as a function of frequency of barium ferrite prepared in aqueous solution and calcined at 800 °C	66
(4-14)	Dielectric constant as a function of frequency of barium ferrite prepared in aqueous solution and calcined at 1000 °C	66
(4-15)	Dielectric constant as a function of frequency of barium ferrite prepared in ethanol/water solution and calcined at 800 °C	67
(4-16)	Dielectric constant as a function of frequency of barium ferrite prepared in ethanol/water and calcined at 1000 °C	67
(4-17)	The variation of loss tangent with frequency of barium ferrite prepared in aqueous solution and calcined at 800 °C	69
(4-18)	The variation of loss tangent with frequency of barium ferrite prepared in aqueous solution and calcined at 1000 °C	70
(4-19)	The variation of loss tangent with frequency of barium ferrite prepared in ethanol/water solution and calcined at 800 °C	70
(4-20)	The variation of loss tangent with frequency of Barium ferrite prepared in ethanol/water solution in and calcined at 1000 °C	71

## LIST OF SYMBOLS AND ABBREVIATIONS

Symbol	Definition	Unit
f	Frequency	Hz
H <sub>c</sub>	Coercivity force	Oe
В	Magnetic Flux density	Т
d	Separated distance	mm
Н	Magnetic field	V/cm
ε <sub>o</sub>	Permittivity of Vacuum (8.85x10 <sup>-12</sup> )	F/m
ε <sub>r</sub>	Relative permittivity (Dielectric Constant)	
C <sub>o</sub>	Vacuum capacitance	F
С	Capacitance of dielectric material	F
3	Permittivity of the dielectric material	F/m
P <sub>ω</sub>	Loss power	W
ł	Thickness	m
V	Volume	m <sup>3</sup>
F	Load handing	kg
А	Area	m <sup>2</sup>
ρ	Resistivity	Ω.m
R	Electrical resistance	Ω
Ø	Electric conductivity	$(\Omega.m)^{-1}$
(hkl)	Miller indices	
V	Voltage	V
Ι	Current	А
X <sub>c</sub>	Impedance of capacitor	Ω
ω	Angular frequency $(2\pi f)$	rad.s <sup>-1</sup>
j	Imaginary route, $\sqrt{-1}$	

φ	Phase angle	degree
δ	Loss angle	degree
I <sub>a</sub>	Active current (resistive current)	А
Ir	Reactive current (capacitive current)	А
$\varepsilon'_r$	Real dielectric constant	
$\mathcal{E}_r''$	Imaginary dielectric constant	
Q	Quality factor	
tanð	Tangent loss angle	
D	Crystallite size	nm
β	Full width at half maximum	rad
θ	Diffraction angle	degree
h	Dielectric thickness	mm
k	Boltzmann constant	$eV.k^{-1}$
Т	Temperature	°C
d	Bulk density	g/cm <sup>3</sup>
λ	Wavelength	Å
W	Mass	g
D	Electrical displacement	C/m <sup>2</sup>

## List of Abbreviations

Symbol	Definition
SEM	Scanning Electron Microscope
XRD	X-ray diffraction
FWHM	Full Width at Half Maximum
H.V	Vickers Hardness
JCPDS	Joint Committee on Powder Diffraction Standards



# Introduction and Previous Studies

#### **1.1 Introduction**

In the last two decades, new terms with the prefix "nano" have rushed into the scientific vocabulary nano particle, nano structure, nanotechnology, nano material, nano cluster, nano chemistry, nano colloids, nano reactor and so on. A series of new journals are devoted particularly to this subject, monographs with the corresponding names have appeared, "nano" specialized institutes, chairs and laboratories have been founded; and numerous conferences are held. In most cases new names are applied to long known objects or phenomena; however, new objects inaccessible to researchers some 20 years ago have also appeared these include fullerenes, quantum dots, nano tubes, nano films and nano wires, i.e., the objects with at least one nanometer  $(10^{-7}-10^{-9}m)$  dimension. The enhanced interest of the researchers in nano objects is due to the discovery of unusual physical and chemical properties of these objects, which is related to the manifestation of the so called "quantum size effects", these arise in the case where the size of the system is commensurable with the de Broglie wavelengths of the electrons, phonons or exciting propagating in them [1].

The research of nano crystalline magnetic materials has undergone a huge development in the last years. This is due to the properties common to both amorphous and crystalline materials and the ability of these alloys to compete with their crystalline counterparts. The benefits found in the nano crystalline alloys stem from their chemical and structural variations on a nano scale which are important for developing optimal magnetic properties. It is well known that the microstructure, especially the crystallite size, essentially determines the hysteresis loop of the soft ferromagnetic materials. The reduction of crystallite size to the dimensions of the domain wall width increases the coercivity towards an extreme value controlled by the anisotropy. However, the lowest coercivity is found again for crystallite smaller than the correlation lengths like in amorphous and nano crystalline alloys [2].

Nanotechnology is the understanding and control of matter at dimensions of roughly 1–100 nm, where unique phenomena enable novel applications. The physical and chemical properties of the nano materials tend to be exceptionally closely dependent on their size and shape or morphology. As a result, materials scientists are focusing their efforts on developing simple and effective methods for fabricating nano materials with controlled size and morphology and hence tailoring their properties. An important aspect of the nano scale is that smaller particle size, larger its surface area. At the nano scale, properties like electrical conductivity and mechanical strength are not the same as they are at bulk size, but changes dramatically. Recently, the synthesis of nano magnetic materials has been a field of intense study, due to the novel microscopic properties shown by particles of quantum dimensions located in the transition region between atoms and bulk solids [3].

Quantum size effects and the large surface area of magnetic particles dramatically change some of the magnetic properties and exhibit superparamagnetic phenomena and quantum tunneling of magnetization, because each particle can be considered as single magnetic domain Magnetic nano particles which have attracted the increasing interest both in fundamental science and in technological applications because of their various useful properties in the recent trend of nanotechnology.

A variation in particle size can also modulate the physical properties even without varying the composition. As the size of the particles decreases below 100 nm, a large fraction of the constituting atoms are found on the surface of the nano crystals; this induces significant changes in the magnetic structure and properties of the materials at nano scale as compared with their bulk counterpart. Specially, the domain wall structure encountered in the bulk crystalline ferrites is replaced by a single domain structure characteristic of each particle, thus leading to new phenomena such as superparamagnetism, extra anisotropy contributions and spin canting [4].

2

Magnetic nano particles of spinel ferrites are of great interest in fundamental science, especially for addressing the fundamental relationships between magnetic properties, their crystal chemistry and structure. Crystal chemistry shows how the chemical composition (chemical formula) which are the internal structure and physical properties of minerals are related each other. Spinel ferrites have been investigated in recent years for their useful electrical and magnetic properties and applications in information storage systems, magnetic bulk cores, magnetic fluids, microwave absorbers and medical diagnostics. The synthesis and magnetic structure characterization of ferrites have been investigated with much interest [4-5] and a lot of attention has been focused on the preparation and characterization of superparamagnetic metal oxide nano particles of spinel ferrites with chemical formula, MeFe<sub>2</sub>O<sub>4</sub> (where Me = Co, Mg, Mn , Zn) etc [6-7].

Nano phase materials with an average grain size in the range of 1 to 50 nm have attracted research interest for more than a decade since their physical properties are quite different from that of their bulk micron-sized counterparts because of the large volume fraction of atoms that occupies the grain boundary area [8-9]. This is a new class of materials which are being used in important applications like high frequency transformers, Ferro fluids, pigments in paints and ceramics, biomedical applications like drug delivery system, hyperthermia, NMR, high density magnetic recording and dye-sensitized solar cells [10-11]. The increase in the interfacial energy due to defects, dislocations and lattice imperfections leads to changes in various physical properties and hence one can tailor make the materials with specific properties. Almost 50 % of the atoms reside in the grain boundary area when the grain size is 100 nm [8-12]. Since a large fraction of atoms is present at the grain boundaries, the nano crystalline materials exhibit enhanced diffusivity.

The magnetic materials are classified into two groups such as soft and hard magnetic materials. Soft magnetic materials have low magneto crystalline anisotropy resulting in reduced coercivity and high permeability such as (Fe-Si-B-Nb-Cu) and (Fe-M-B-sCu) alloys. The high Curie temperature (Fe-Co- Cu-Zr) alloys are used in the high application. The saturation magnetization for the nano crystalline ferrites, in general, is found to be lower compared to their bulk value, which is attributed to surface spin effects. In some cases an enhancement in the saturation magnetization is observed due to the change in cation distribution which depends on the crystal field stabilization energy of the cations . Apart from grain size, the cation distribution, which depends on the synthesis condition, is found to play a major role in the observed changes in their magnetic properties. The high coercivity of the hard magnetic materials arises from the large magneto crystalline anisotropy of the materials with a non-cubic structure. The nano magnetic materials can be synthesized with varying grain size using the techniques like mechanical milling ,sol gel method, laser ablation method, oxidation method, reverse micelle polyol process and co precipitation method etc.

#### **1.2 Previous Studies**

Magnetism is very common to everything you see. The term magnetism comes from a rock called lodestone. The ancient Greeks and the Chinese knew about this strange and rare stone with the power to attract iron This natural form of magnet was then named as lodestone. The name magnet was used by the Greeks for this lodestone, because of its property of attracting other pieces of the same material and iron as well. It has been proved later that this naturally occurring lodestone is the magnetic iron oxide or the naturally occurring mineral called magnetite. The Chinese found that when steel needle stroked with such a lodestone became magnetic and the needle pointed north -south when it is suspended freely. They applied this characteristic feature of the lodestone for navigation, fortune telling and a guide for building. Some of the properties of magnets were discovered earlier than 600 RC, although it is only in the twentieth century that physicists have begun to understand why substances behave

magnetically. Thus magnetism is one of the earliest known physical phenomena of solid materials.

In 2005, Nanocrystalline structure and magnetic properties of barium ferrite particles have been studied by **Jianxun Qiu** etal, using a self propagating combustion method with glycine as a fuel. The process investigated with differential scanning calorimeter and thermogravimetric analysis. The effects of the pH value of the precursor solution, the glycine dosage and calcination temperature on the morphology, the crystalline structure and the magnetic properties of the barium ferrite particles were studied by means of X-ray diffraction, infrared spectra, field emission scanning electron microscopy and vibrating sample magnetometry. The results show that the ignition temperature of glycine gel is lower than that of citric acid gel. The pH value of the precursor solution and calcination. Barium hexaferrite particles with a specific saturation magnetization of  $56.1 \text{Am}^2/\text{kg}$  and a coercivity of 398.0kA/m obtained when the pH value of the precursor solution was 7.0 and the calcination temperature was  $850 \,^{\circ}\text{C}$  [13].

In **2005**, The researchers **Jozef Sláma** etal, studied properties of M-type barium ferrite doped by selected ions . The magnetic and structural properties of substituted Ba hexaferrite M-type samples with composition  $BaFe_{12-2x}(Me_1-Me_2)_xO_{19}$ , where  $Me_1 = Zn$ , Co, Ni, and  $Me_2 = Zr$ , Ti, were compared. The powder samples with  $(0.0 \le x \le 0.6)$  were prepared by two processing routes. Different Fe/Ba ratio was used for mechanical alloying (Fe/Ba = 10.0) and for the citrate precursor method (Fe/Ba = 10.8), Magnetic properties studied by both vibrating sample magnetometry and thermo magnetic analysis. The ferrite formation process followed by Mssbauer spectroscopy [14].

In **2006**, the researcher **D Bahadur** etal, synthesize single-domain barium ferrite nano particles with narrow particle size distribution using an auto combustion technique. In this process, citric acid was used as a fuel. Ratios of cation to fuel were maintained variously at 1: 1, 1: 2 and 1: 3. The pH was 7 in

all cases. Of all three cases, a cation to citric acid ratio of 1:2 gives better yield in the formation of crystalline and single domain particles with a narrow range of size distribution. Most particles are in the range of 80 to 100 nm. Maximum magnetization and coercivity values are also greater for 1 : 2 ratios. These values measured at room temperature are found to be 55 emu / gram and 5000 Oe respectively. XPS and ESR studies support the result [15].

in **2007**, Chemical co-precipitation methods have been used to synthesis Hexagonal ferrite powder by **Hsing-I Hsiang**, **Ren-Qian Yao**, and it was found that the BaM phase was formed directly through the reaction of the preceding  $\alpha$ -Fe<sub>2</sub>O<sub>3</sub> and amorphous BaCO<sub>3</sub> for BaM precursor. For the Co<sub>2</sub>Y precursor, the intermediate phase, BaM, was obtained through the reaction of the earlier formed BaFe<sub>2</sub>O<sub>4</sub> and  $\alpha$ -Fe<sub>2</sub>O<sub>3</sub>. The Co<sub>2</sub>Y phase has been obtained through a BaM and BaFe<sub>2</sub>O<sub>4</sub> reaction. However, for the Co<sub>2</sub>Z precursors, the BaM phase was obtained directly from the BaCO<sub>3</sub> and amorphous iron hydroxide reaction, with no  $\alpha$ -Fe<sub>2</sub>O<sub>3</sub> and BaFe<sub>2</sub>O<sub>4</sub> formed as intermediates. Co<sub>2</sub>Z phase was obtained through the reaction of the two previous formed BaM and Co<sub>2</sub>Y phases [16].

In 2007, Ping Xu etal, have synthesized BaFe<sub>12</sub>O<sub>19</sub> hexaferrite nanoparticles, containing cetyltrimethylammonium chloride (CTAC). n-hexanol, and cyclohexane, using a reverse microemulsion technique, with a combination of (NH<sub>4</sub>)<sub>2</sub>CO<sub>3</sub> and NH<sub>3</sub>H<sub>2</sub>O as precipitator. Barium ferrite nanoparticles with 30 nm diameter and uniform flaky structure were proved to be single magnetic domains, which have magnetic properties comparable to some of the best ever reported for fine barium ferrite powders by chemical methods. Heat-treatment conditions can significantly influence the formation of pure BaFe<sub>12</sub>O<sub>19</sub> hexaferrite phase, where quenching and nonprecalcination would produce intermediates of R-Fe<sub>2</sub>O<sub>3</sub> and BaFe<sub>2</sub>O<sub>4</sub>, as detected by X-ray diffraction (XRD) and Fourier transform infrared (FTIR) analyses, resulting in lower magnetic properties. High magnetocrystalline anisotropy constant K and energy barrier  $E_A$  calculated from Stoner-Wohlfarth

theory may also account for the high coercivity for pure  $BaFe_{12}O_{19}$ . The variation of electrical conductivity during the formation and reaction of microemulsion droplets suggests nonpercolating microemulsion conducting systems. Transmission electron microscopic (TEM) images of the microemulsion droplets from a microemulsion system with water to oil ratio of 1:8 displayed microemulsion droplets about 100 nm, containing a barium ferrite precursor "core" of about 30 nm in size, with collision and coalescence being discovered [17].

In 2007, R. Nowosielski etal, prepared barium ferrite powder by milling and heat treatment (annealing). The milling process was carried out in a vibratory mill, which generated vibrations of the balls and milled material inside the container during which their collisions occur. After milling process the powders were annealed in electric chamber furnace. The X-ray diffraction methods were used for qualitative phase analysis of studied powder samples. The distribution of powder particles was determined by a laser particle analyzer. The magnetic hysteresis loops of examined powder material were measured by resonance vibrating sample magnetometer (R-VSM). The milling process of iron oxide and barium carbonate mixture causes decrease of the crystallite size of involved phases. The X-ray investigations of tested mixture milled for 30 hours and annealed at 950°C enabled the identification of hard magnetic BaFe<sub>12</sub>O<sub>19</sub> phase and also the presence of  $Fe_2O_3$  phase in examined material. The  $Fe_2O_3$  phase is a rest of BaCO<sub>3</sub> dissociation in the presence of Fe<sub>2</sub>O<sub>3</sub>, which forms a compound of  $BaFe_{12}O_{19}$ . The best coercive force (H<sub>C</sub>) for mixture of powders annealed at 950 °C for 10, 20 and 30 hours is 349 kA/m, 366 kA/m and 364 kA/m, respectively. The arithmetic mean of diameter of Fe<sub>2</sub>O<sub>3</sub> and BaCO<sub>3</sub> mixture powders after 30 hours of milling is about 6.0 µm. The barium ferrite powder obtained by milling and annealing can be suitable components to produce sintered and elastic magnets with polymer matrix. The results of tested barium ferrite investigations by

different methods confirm their utility in the microstructure and magnetic properties analysis of powder materials [18].

In **2010**, **A.Ataie and S. Jahangir** prepared nano size particle of barium hexaferrite by conventional mixed oxide ceramic method from  $BaCO_3$  and  $\alpha$ -Fe<sub>2</sub>O<sub>3</sub> as starting materials . 3% wt. NaCl was used as a process control agent (PCA) and influence of the intensive milling has been investigated .Thermal analysis, phase composition, morphology and magnetic properties of the products were characterized using DTA/TG, XRD, SEM/TEM and VSM techniques , respectively . In all milled samples  $BaCO_3$  were completely decomposed. Formation temperature of barium hexaferrite decreased in sample milled with NaCl for 10 h .NaCl is considered to be efficient in reduction of crystallite size and acceleration of barium hexaferrite reaction kinetic. Mean crystallite size of the barium hexaferrite in sample milled for 10 h in the presence of NaCl and annealed at 1000°C was measured 25 nm using TEM images. Hc and Ms in the above sample were 4.3 kOe and 52.7 emu/g , respectively [19].

In **2010, H.Z. Wanga** etal, prepared Barium hexaferrite (BaFe<sub>12</sub>O<sub>19</sub>) with hexagonal structure, by sintering the mixture of  $\alpha$ -Fe<sub>2</sub>O<sub>3</sub> and BaCO<sub>3</sub>, ball milling of the mixture followed by heat treatment as well as glycine-nitrate method and subsequent heat treatment, respectively. The mechanism of formation of the BaFe<sub>12</sub>O<sub>19</sub> in the three kinds of procedures investigated by using sintering shrinkage curve and XRD measurements. It was found that the  $\alpha$ Fe<sub>2</sub>O<sub>3</sub> reacted with BaCO<sub>3</sub> to form BaFe<sub>2</sub>O<sub>4</sub> with orthorhombic structure as the mixture were sintered above 660 °C firstly and then the BaFe<sub>2</sub>O<sub>4</sub> reacted with  $\alpha$ Fe<sub>2</sub>O<sub>3</sub> to form BaFe<sub>12</sub>O<sub>19</sub> in a sintering temperature ranging from 770 to 920 °C. However, the  $\alpha$ -Fe<sub>2</sub>O<sub>3</sub> reacted with BaCO<sub>3</sub> to form Ba<sub>x</sub> Fe<sub>3-x</sub> O<sub>4</sub> with spinel structure when the mixture was milled for 80 h, while the BaFe<sub>12</sub>O<sub>19</sub> was obtained by annealing the Ba<sub>x</sub>Fe<sub>3-x</sub>O<sub>4</sub> at 700–1000 °C. In the glycine-nitrate procedure, the precursor powders containing  $\alpha$ Fe<sub>2</sub>O<sub>3</sub> , Fe<sub>3</sub>O<sub>4</sub> and BaFe<sub>2</sub>O<sub>4</sub> were fabricated by self propagating reaction firstly, and then the single BaFe<sub>12</sub>O<sub>19</sub> was produced by sintering the precursor powders at 1000 °C. The magnetic properties of the  $BaFe_{12}O_{19}$  produced by the three kinds of procedures were reached. The saturation magnetization and the coercivity of the  $BaFe_{12}O_{19}$  fabricated by ball milling followed by heat treatment were 47.24emu/g and 5086.34 Oe, respectively, which were much larger than those of the  $BaFe_{12}O_{19}$  produced by other procedures [20].

In 2010, the barium ferrite particles have been prepared by P M Prithvira Swamy etal, using a self-propagating low-temperature combustion method with polyethylene glycol (PEG) as fuel. The process was investigated with simultaneous thermo gravimetric- differential thermal analysis (TG–DTA). The crystalline structure, morphology and the magnetic properties of the barium ferrite particles are studied by means of X-ray diffraction (XRD), scanning electron microscopy (SEM), transmission electron microscopy (TEM) and SQUID susceptometer. The results show that the ignition temperature of PEG is lower compared with other combustion methods and gives nano crystalline barium ferrite [21].

In **2010**, **H** . Ovalioglua etal, were synthesized  $Ba_{1-x}La_x Fe_{12}O_{19}$ 

 $(0 \le x \le 0.4)$  and BaFe<sub>12-y</sub>Nd<sub>y</sub>O<sub>19</sub> ( $0 \le y \le 0.4$ ) samples in ammonium nitrate melt and nitric acid by different synthesis route .The effects of La and Nd doping on the microstructure and magnetic properties of BaFe<sub>12</sub>O<sub>19</sub> were investigated. The highest coercive field was obtained for the sample having La content, x = 0.3[22].

In **2010**, **M.A Iqbal** etal, synthesized nanosized barium hexaferrites ,using solgel combustion method . samples of different composition with varying molar ratio of metal nitrates and citric acid of (0.5, 1, 1.5, 2, 3) were prepared. Pellets of powders of all samples were structurally characterized by X-ray diffractometer. Lattice parameter, volume of unit cell and particle size was determined. It has been found that size of the particle increases by decrease in the citric acid content. It was limited the particle size calculated by debey Sherrer formula . Resistivities of specimens indicate semiconducting behavior . Measured values of resistivity are well in agreement with standard barium ferrite [23].

In **2010**, Barium hexaferrite has been synthesised by **Q.Mohsen** by Effect of different annealing temperature on the particle size, microstructure and magnetic properties of the resulting barium hexaferrite powders has been studied, and reported in the presented paper. The  $Fe^{3+}/Ba^{2+}$  mole ratio is controlled at 12, while the annealing temperature was controlled from 800 to 1200 °C. The resultant powders were investigated by differential thermal analyzer (DTA), X ray diffractometer (XRD), scanning electron micros-copy (SEM) and vibrating sample magnetometer (VSM). Single well crystalline  $BaFe_{12}O_{19}$  phase was obtained at annealing temperature 1200 °C. The SEM results show that the grains were regular hexagonal platelets. In addition, maximum saturation magnetization (66.36 emu/g) was observed at annealing temperature 1100 °C. However, it was found that the coercivity of the synthesized  $BaFe_{12}O_{19}$  samples was lower than the theoretical values [24].

In **2010, A.M.Bhavikatti** etal, synthesized nano-sized barium ferrite (BaFe<sub>2</sub>O<sub>4</sub>), by using microwave route. The sample was characterized by X-ray diffraction, SEM and IR studies. Electrical resistance(R) measurements of the sample revealed a very high resistivity and the results indicated a fall in resistivity with the rise in temperature. The obtained dc conductivity is found to be very low of the order of  $10^{-8}$  S/cm. The dc conductivity follows the Arrhenius law. The hysteresis M-H loops for this sample was traced using the vibrating sample Magnetometer (VSM) to study the parameters coercivity (Hc), saturation magnetization (Ms), remanence (Mr) and squareness ratio (SQR). The complex impedance spectroscopy (CIS) technique was used to study the electrical response of the nano structured sample in a wide range of frequencies (1 KHz to 1MHz) at different temperatures (0–700°C) [25].

In **2011**, the researchers **Jiajie Liu** etal, prepared barium ferrite-containing glass ceramic fibers, by the combination of a sol–gel process and electrospinning

technique using basic iron formate, barium acetate and boric acid as the starting materials. After leaching of barium borate matrix, pure phase  $BaFe_{12}O_{19}$  fibers obtained. The relationship of aged time and viscosity of the precursor solution was studied and the results showed that the viscosity corresponding to the spinnable state is 1–4 Pa s. The morphology, structure and magnetic properties of the obtained fibers were characterized with scanning electron microscopy, transmission electron microscopy, fourier transform infrared spectroscopy, thermo gravimetric analysis–differential scanning calorimetry, vibrating sample magnetometer. The X-ray diffraction results indicate that only the M-type Baferrite and Ba-borate exist. The fibers had rough surface and hollow structure with the diameter no more than 1 µm. The fibers were composed of 40 nm  $BaFe_{12}O_{19}$  nanoparticles embedded in the borate matrix. The coercivity and saturation magnetization of the synthesized fibers were 4,106.9 Oe and 17.8 emu/g, respectively [26].

In **2011**, the nanocrystalline barium hexaferrite (BaFe<sub>12</sub>O<sub>19</sub>) powders have been synthesized using the sol gel auto combustion method by **Widyastuti** etal, The ferrite precursors were obtained from aqueous mixtures of barium nitrate and ferric nitrate by auto combustion reaction from gel point. These precursors were sintered at different temperatures ranging from 700 to 1000°C for constant calcinations time of 2.5 h in a static air atmosphere. Effects of Fe<sup>3+</sup>/Ba<sup>2+</sup> mole ratios and sintering temperatures on the microstructure and magnetic properties were systematically studied. The powders formed were investigated using X-ray diffraction (XRD), scanning electron microscope (SEM) and VSM. The results obtained showed that the phase BaFe<sub>12</sub>O<sub>19</sub> powders achieved by the Fe<sup>3+</sup>/Ba<sup>2+</sup> mole ratio from the stoichiometric value 11, 11.5 and 12 at temperature 950°C. With increasing of temperature sintering, coercivity and magnetization value tends to rise. The maximum saturation magnetization (66.16 emu/g) was achieved at the Fe<sup>3+</sup>/Ba<sup>2+</sup> mole ratio to 11.5 and the sintering temperature of 950°C. The maximum coercivity value 3542 Oe achieved at mole ratio being 12 with

sintering temperature 950°C. Maximum saturation 6616 emu/g achieved at mole ratio of 115 at same temperature[27].

In **2011**, Barium hexaferrite (BaFe<sub>12</sub>O<sub>19</sub> - BaF) powder was synthesized , by **S.Kanagesana** etal, using a sol-gel combustion route using metal nitrates and D-Fructose. BaFe<sub>12</sub>O<sub>19</sub> precursor was calcined using microwave furnace at 900°C for 10 minutes. The products characterized by X-ray diffraction (XRD), high resolution scanning electron microscope (HR-SEM), and vibrating sample magnetometer (VSM), whose results indicate that they have well crystalline phase of barium hexaferrite, the diameter of the hexagonal platelet-like structure ranges from 50 to 100 nm. Magnetic measurements show that the coercivity was 5558.6 Oe and saturation magnetization 55.20 emu/g. Magnetic measurements for the barium hexaferrite showed enhanced coercivity [28].

In 2011, Microemulsion processing and co-precipitation technique was used, by Virk H. S. etal, to prepare  $BaFe_{12}O_{19}$  hexaferrite precursors containing cetyl trimethyl ammonium bromide (CTAB). The prepared barium hexaferrite precursors calcined at 950°C for 4 hours in a furnace, then slowly cooled to room temperature. The effect of synthesis techniques on structural and magnetic hexaferrites has been studied. Decomposition behavior is properties of investigated by means of thermal analysis (TGA). Fourier Transform Infrared (FTIR) and X-ray diffraction (XRD) techniques have been used for characterization of barium hexaferrite particles. The X-ray diffractogram of the sample prepared by using a co-precipitation technique and microemulsion process show only single M-phase. Surface morphology of nonporous particles was examined using SEM and TEM. Magnetic measurements were carried out at room temperature using a Vibration Sample Magneto-meter (VSM). The maximum coercivity and saturation magnetisation of the prepared BaFe<sub>12</sub>O<sub>19</sub> hexaferrite samples are determined by a VSM. The value of saturation magnetisation depends on the type of synthesis technique used. The sample prepared by a microemulsion route shows high saturation magnetisation (~71

emu/g), where as the sample prepared by a co-precipitation route exhibits low saturation magnetisation (~65 emu/g). Magnetic study reveals that prepared BaFe12O19 hexaferrite particles using both techniques possess single magnetic domain [29].

In **2012**, microwave-hydrothermal method was used by **K. Sadhana** etal, to prepare BaFe<sub>12</sub>O<sub>19</sub> nanocrystalline at 200 °C/ 45 min. The as-synthesized powders were characterized by using X-ray diffraction (XRD), thermogravimetry (TG) and differential thermal analysis (DTA). The present powders were densified at different temperatures, i.e., 750, 850, 900 and 950 °C for 1 h using microwave sintering method. The phase formation and morphology studies were carried out using XRD and field emission scanning electron microscopy (FE-SEM). The average grain sizes of the sintered samples were found to be in the range of 185–490 nm. The magnetic properties such as saturation magnetization and coercive field of sintered samples were calculated based on magnetization curves. A possible relation between the magnetic hysteresis curves and the microstructure of the sintered samples was investigated [30].

In **2012**, **M.J. Molaei** etal, prepared mixtures of barium ferrite, and graphite with different molar ratios and mixed and milled in a planetary ball mill to produce a BaFe<sub>12</sub>O<sub>19</sub>/Fe<sub>3</sub>O<sub>4</sub> nano-composite. Some of the milled samples were then heat treated at 750 and 850 °C under vacuum to synthesize BaFe<sub>12</sub>O<sub>19</sub>/Fe/Fe<sub>3</sub>O<sub>4</sub> composite. The effects of graphite content in the mixtures (C:O molar ratio of 0.9, 1 and 1.1), milling time and heat treatment on the characteristics of powder mixtures were investigated by XRD, VSM and TEM. XRD results showed that barium ferrite was partially reduced to Fe<sub>3</sub>O<sub>4</sub> during milling. It was also found that increasing the C:O molar ratio from 0.9 to 1.1 accelerated the reduction process. The results show that the magnetic properties of the milled and heat treated samples have been altered by changing the C:O molar ratio .The saturation magnetization of BaFe<sub>12</sub>O<sub>19</sub>/Fe<sub>3</sub>O<sub>4</sub> nano-composite formed in the 40 h milled sample with a C:O molar ratio of 0.9 was 27.86 emu/g. This value increased to 156.40 emu/g after heat treatment at 850 °C. Increasing the C:O molar ratio from 0.9 to 1.1 increased the saturation magnetization to 169.19 emu/g in the heat treated sample. The VSM results also showed that the coercivity of milled and heat treated samples is influenced by C:O molar ratio. The lowest coercivity value of 24.36 Oe was obtained in the sample milled for 40 h and then heat treated at 850 °C. This result is correlated well with the XRD results and confirmed mainly the soft magnetic nature of the produced nano-composite [31].

In **2012, Cong-Ju Li** etal, prepared  $Ba_{(1 x)}La_xFe_{12}O_{19}$  (0.00  $\le x \le 0.10$ ) nano fibers. It was fabricated via the electro spinning technique followed by heat treatment at different temperatures for 2 h. Various characterization methods including scanning electron microscopy (SEM), X-ray diffraction (XRD), vibrating sample magnetometer (VSM), and microwave vector network analyzer were employed to investigate the morphologies, crystalline phases, magnetic properties, and complex electromagnetic parameters of nano fibers. The SEM images indicate that samples with various values of x are of a continuous fiberlike morphology with an average diameter of  $110 \pm 20$  nm. The XRD patterns show that the main phase is M-type barium hexaferrite without other impurity phases when calcined at 1100 °C. The VSM results show that coercive force  $(H_c)$ decreases first and then increases, while saturation magnetization (M<sub>s</sub>) reveals an increase at first and then decreases with La<sup>3+</sup> ions content increase . Both the magnetic and dielectric losses are significantly enhanced by partial substitution of  $La^{3+}$  for  $Ba^{2+}$  in the M-type barium hexaferrites . The microwave absorption performance of Ba<sub>0.95</sub> La<sub>0.05</sub> Fe<sub>12</sub> O<sub>19</sub> nano fibers gets significant improvement : The bandwidth below -10 dB expands from 0 GHz to 12.6GHz, and the peak value of reflection loss decreases from

-9.65 dB to -23.02 dB with the layer thickness of 2.0 mm [32].

### 1.3 Aims of the Study

**1.** Preparing barium ferrite from barium chloride and ferric chloride and sodium hydroxide.

**2.** Characterization of the prepared Barium Ferrite using XRD, SEM, Vickers device and LCR meter.

**3.** Studying some factors (Density, Hardness, Dielectric constant, Electrical resistivity, Tangent loss and Electrical conductivity) and the influence of the effecting factors (temperature and frequency) on them.



# Theoretical Background

#### 2.1 Introduction

This Chapter reviews the theoretical part which includes the explanation of nanotechnology and the basic concepts of permanent magnetic materials and importance on M-type ferrite particularly barium ferrite permanent magnets. Mtype ferrites are compared with other important permanent magnets and their historical development is described. The crystal structure, magnetic structure and phase relationship of barium ferrite are outlined. The intrinsic magnetic properties of M-type ferrite are presented in details. An account of the various processing techniques used for manufacturing. Finally, the properties of commercial barium ferrite permanent magnets and applications are also illustrated.

#### 2.2 Nanotechnology

One of the most exciting and fastest growing area in science and engineering today is nanotechnology, materials science and electronics to create new function systems of nanoscale dimensions of the order of a billionth of a meter, nanotechnology refers to the study manipulation and utilization of nano size materials (sizes of atoms and molecules) [2].

Nanotechnology defines a particle as a single unit in terms of its properties and transport. Assuming a particle to be roughly spherical, classification can be done on the basis of their diameters. Fine particles are those which have sizes in the range of 100 to 2500 nm. Individual molecules, even though having sizes that would normally lie within the aforementioned ranges cannot be considered as nanoparticles. Nanopowders are nanoparticles in an agglomerated form. They have nano-scale dimensions in all the three directions.

In nanomaterials, a majority of the atoms are present on the surface as opposed to conventional materials where the atoms are found to be present in the bulk as well. Owing to the presence of atoms in diverse environments, nanopowders have intrinsic properties different from those of conventional materials. Nanopowders have high surface areas, so their properties are largely

16

augmented. The characteristic high surface area of nanopowders enhances the rate of densification. Sintering of nanopowders can thus take place at a lower temperature [33].

In the recent years, nanopowders have enjoyed a great deal of attention, because Perovskite nanopowders, in particular have been thoroughly investigated for their excellent dielectric properties. These powders are found to be good candidates for gas sensors. The tremendous usage of perovskite nanopowders in these fields stems from the fact that their properties can be easily modified by selecting a particular cation. These powders also have excellent thermal stability [34].

The progress of technology and quality of life of mankind have always been closely knit with the progress in material science and material processing technology. Most material processing techniques are based on breaking up a large chunk of material into desired shapes and sizes, inducing strain, lattice defects and other deformations in the processed material. Recent developments in nanotechnology and the demonstration of various quantum size effects in nanoscale particles imply that most of the novel devices of the future will be based on the properties of nanomaterials, each nanoparticle contains only about 3-10<sup>7</sup> atoms/molecules. Lattice defects and other imperfections induced by the traditional material processing techniques will no longer be diluted by sheer number of atoms, when used for synthesizing nanoparticles. Furthermore, it is difficult to achieve size selective synthesis of such small particles, by using the traditional approach. The alternative synthetic technique for nanoparticles involves controlled precipitation of nanoparticles from precursors dissolved in a solution [35].

Nanoparticle preparation and study of nanoparticle are important in the recent research. The characteristics of metal nano particles like optical, electronic, magnetic, and catalytic depend on their size, shape and chemical

17

surroundings . In nanoparticle preparation, it is very important to control the particle size, particle shape and morphology [36].

Nanomaterials and nanoparticles are used in a broad spectrum of applications. Today they are contained in many products and use in various technologies. Most nanoproducts produced on an industrial scale are nanoparticles, although they also arise as by products in the manufacture of other materials.

#### 2.3 Production of nanoparticles and nanomaterials

Two basic strategies are used to produce nanoparticles: "top-down" and "bottom -up". The term "top-down" refers here to the mechanical crushing of source material using a milling process [37]. In the "bottom-up" strategy, structures are built up by chemical processes . The selection of the respective process depends on the chemical composition and the desired features specified for the nanoparticles, this is shown in figure(2-1).



#### Figure (2-1): Methods of nanoparticle production [38].

"Top-down" refers to mechanical-physical particle production processes based on principles of microsystem technology. The traditional mechanical-physical crushing methods for producing nanoparticles involve various milling techniques [37]. This method does not allow full control of particle shape and is shown in Figure (2-2).



Figure (2-2): Mechanical-physical nanoparticle production processes [38]. Bottom-up methods are based on physicochemical principles of molecular or atomic self-organization. This approach produces selected, more complex structures from atoms or molecules, better controlling of shapes and size ranges. It includes aerosol precipitation reactions and sol-gel processes [37].

#### 2.4 Properties of nanoparticles

Nanoparticles are of interest because of surface properties such as chemical, electrical, optical, magnetic and mechanical; begin to dominate over the bulk material. Incorporation of these 'designer' particles into other materials, exploiting the properties of the nano particle surface atoms, allowing the formation of novel products with enhanced or entirely different properties. Metals can be made stronger and harder; ceramics can have enhanced ductility and formability, normally insulation of materials can be made to conduct heat or electricity, and protective coatings can become transparent [39].

#### 2.5 Precipitation processes

The precipitation of solids from a metal ion containing solution is one of the most frequently employed production processes for nanomaterials. Metal oxides as well as non-oxides or metallic nanoparticles can be produced by this
approach, the process is based on reactions of salts in solvents. A precipitating agent is added to yield the desired particle precipitate, and the precipitate is filtered out and thermally post-treated. In precipitation processes, particle size, size distribution, crystallinity and morphology (shape) are determined by reaction kinetics (reaction speed). The influencing factors include, beyond the concentration of the source material, the temperature, pH value of the solution, the sequence in which the source materials are added, and mixing processes [38]. Chemo-physical methods is shown in figure (2-3).



Figure (2-3): Chemo-physical processes in nanoparticle production [38].

### 2.6 Ferrite and Ferrite's Structure

Ferrites are ceramic, homogeneous materials composed of various oxides with iron oxide as their main constituent. Ferrites can have several distinct crystal structures. Many of them are magnetic materials and they are used to make permanent magnets, ferrite cores for transformers, and in various other applications. Ferrites are usually nonconductive ferrimagnetic ceramic compounds derived from iron oxides such as hematite (Fe<sub>2</sub>O<sub>3</sub>) or magnetite (Fe<sub>3</sub>O<sub>4</sub>) as well as oxides of other metals, and are like most other ceramics, hard and brittle. In terms of their magnetic properties, the different ferrites are often classified as "soft" or "hard", which refers to their low or high magnetic coercivity [40]. Ferrite has a three-dimensional, crystalline structure. Its chemical formula is MO.Fe<sub>2</sub>O<sub>3</sub>, which means that it has two atoms of iron and three atoms of oxygen. The MO refers to other metal oxides which could be manganese, zinc or nickel oxide, by adding the other metal oxides you are able to change the property of ferrite to make it suitable for various applications. Ferrite is a material that can be magnetized easily and can hold this property indefinitely even there is an ability to magnetize ferrite so that it has multiple poles [41].

### 2.7 Magnetic materials types

Magnetic materials can be divided into two groups soft and hard magnetic materials. The soft magnetic materials are those materials which are magnetized and demagnetized easily while the hard magnetic materials are those which are difficult to magnetize and demagnetize.

The hard magnetic materials have high coercivity, because the high coercivity resists the magnetization action. The basic difference of two types of permanent magnets was described on the basis of hysteresis loop. The soft magnetic materials exhibit a narrow hysteresis loop, whereas; the hard magnetic materials show a broad hysteresis loop. In the narrow hysteresis loop magnetization follows the variation of the applied field without significant loss. The broad hysteresis loop shows the magnetic energy that can be stored in the materials [42].

### 2.7.1 Soft magnetic materials

Soft magnetic materials can be easily magnetized and demagnetized. They retain their magnetization only in presence of a magnetic field. They show a narrow hysteresis loop, so that the magnetization follows the variation of applied field nearly without hysteresis loss [43]. They are used to enhance the flux, produced by an electric current in them. The quality factor of a soft magnetic material is to measure of its permeability with respect to the applied magnetic field. The other main parameter is the coercivity , saturation magnetization and the electrical conductivity. An ideal soft magnetic material would have low coercivity ( $H_c$ ), a very large saturation magnetization ( $M_s$ ), zero remanence ( $B_r$ ), zero hysteresis loss and very large permeability [44]. The hysteresis loop of soft magnetic material is shown in figure (2-4) [45]. Few important soft magnetic materials are Fe, Fe-Si alloys, soft ferrites (MnZnFe<sub>2</sub>O<sub>4</sub>), silicon iron etc.

### 2.7.2 Hard magnetic materials

Hard Magnetic materials also called as permanent magnets are used to produce strong field without applying a current to coil. Permanent magnets required high coercivity, so they should exhibit a strong net magnetization and is stable in the presence of external fields, which requires high coercivity. In hard magnetic materials uniaxial magnetic anisotropy is necessary and the following magnetic properties are required [46]. The hysteresis loop of hard magnetic material is shown in figure (2-4). Important properties of hard magnetic materials are mentioned below:

**High coercivity:** The coercivity, also called the coercive field, of a ferromagnetic material is the intensity of the applied magnetic field required to reduce the magnetization of that material to zero after the magnetization of the sample has been driven to saturation. Coercivity is usually measured in Oersted or ampere/meter units and is denoted  $H_c$ .

Materials with high coercivity are called hard ferromagnetic materials, and are used to make permanent magnets [47].

**Large magnetization:** The process of making a substance temporarily or permanently magnetic, as by insertion the material in a magnetic field.

**Rectangular hysteresis loop:** A hysteresis loop shows the relationship between the induced magnetic flux density (B) and the magnetizing force (H). hard magnetic materials have rectangular hysteresis loop [48].

22

Hard magnetic material has the wide hysteresis loop due to strong magnetization which is shown in figure (2-4):



Figure (2-4): Hysteresis loop for soft and hard magnetic materials

Hard ferrites have a hexagonal structure and can be classified as M-, W-, X-, Y-, Z- type ferrites [49]. Table (2-1) shows the types of ferrites and their respective chemical formulae:

Types	Chemical Formula	
M-	RFe12O19	R = Ba, Sr, Pb
		2 2 2
W-	RMe <sub>2</sub> Fe <sub>16</sub>	$Me = Fe^{-2} Ni^{-2}, Mn^{-2}etc$
Х-	RMeFe <sub>28</sub>	
Y-	R2Me2Fe12O22	
Z-	R3Me2Fe12O41	

Table (2-1): A	comparison	of the Hard	Ferrites [	[49-50-51]
----------------	------------	-------------	------------	------------

W-, X-, Y-, Z- types are not important economically because of their relatively difficult processing. The chemical composition of various hexagonal compounds is shown in the figure (2-5) [52], as part of ternary phase diagram for BaO-MeO-Fe<sub>2</sub>O<sub>3</sub> system. Here, MeO represents a divalent ion among the first transition elements, Zn, Mg or a combination of ions whose valencey is two. S denotes a cubic spinel MeO.Fe<sub>2</sub>O<sub>3</sub>.



Figure (2-5) Compositional phase diagram for hexagonal ferrites

### 2.8 M-Type ferrites

M-type ferrites with the formulae of BaO.6Fe<sub>2</sub>O<sub>3</sub> (BaM), SrO.6Fe<sub>2</sub>O<sub>3</sub> (SrM) and PbO.6Fe<sub>2</sub>O<sub>3</sub> (PbM) are by far the most important hexagonal ferrites. M-type ferrites are mainly used as permanent magnet materials that have strong resistance to demagnetizing field once they get magnetized and have a dominant position in permanent magnet market. They are preferred over alnicos due to lower material and processing cost and superior coercivity. Sr-Ferrite and Ba-Ferrite are the two main materials in the M-type ferrite family. These ferrites have moderate magnetic properties, and price per unit of available magnetic energy is very low [53].

# 2.9 Intrinsic magnetic properties of M-Type ferrites

The intrinsic magnetic properties are subdivided into primary and secondary one. The primary properties such as saturation magnetization  $J_s$  and magneto crystalline anisotropy constant K1 are directly related to the magnetic structure. The secondary magnetic properties such as anisotropy field strength  $H_A$  and the specific domain wall energy ( $\gamma_w$ ) are derived from the primary ones. The secondary magnetic properties characterize the actual magnetic state. These govern the actual magnetic behavior. The primary and secondary magnetic properties characterize the actual magnetic state. These govern the actual magnetic behavior. The primary and secondary magnetic properties characterize the actual magnetic state. These govern the actual magnetic behavior. The primary and secondary magnetic properties are shown in table (2-2) [53] :

Primary Properties				
Saturation Magnetization, mT	475			
Anisotropic constant, kJ/m <sup>3</sup>	360			
Curie temperature, K	750			
Secondary	Properties			
Specific wall energy, J/ m <sup>2</sup>	54.2 x 10 <sup>-4</sup>			
Anisotropy Field H <sub>A</sub> , kA/m	1506			
Max Coercivity, (H <sub>c</sub> )max	1240			

Table (2-2) Primary and secondary properties of M-Type ferrites

The saturation magnetization, Js is the maximum magnetic moment per unit volume per gram. It is easily derived from the spin configuration of the sublattices, eight ionic moments and 40µB per unit cell, which corresponds to 668 mT at 0K [52].

## 2.10 Structural Properties of ferrites

X-ray diffraction has long been used to determine the atomic scale structure of materials. The technique is based on the fact that the wavelength of X-rays is comparable in size to the distances between atoms in condensed matter. Thus, when a bulk material that exhibits a long-range, periodic atomic order, such as a crystal, is irradiated with X-rays, it acts as an extended, almost perfect grating and produces a diffraction pattern showing numerous sharp spots, called Bragg diffraction peaks. By measuring and analyzing the positions and intensities of Bragg peaks, it is possible to determine the spatial characteristics of the grating, i.e. to determine the three-dimensional (3D) atomic arrangement in bulk crystals. This is the essence of the so-called "crystal structure" [54].

## 2.10.1 Debye-Scherrer-Method

The Debye-Scherrer method uses a crystalline powder and illuminates it with monochromatic X-rays. The diffracted signal is detected by a Geiger-Muller-Tube. The powder sample contains small single-crystals, so-called crystallites. A set of parallel lattice planes (hkl) in a crystallite leads to a constructive interference of reflected X-rays if its alignment fulfills the Bragg condition (2-1) [55].

 $n\lambda = 2d \sin\theta.....(2-1)$ 

Where  $\lambda$ - wavelength of X-ray, d-inter planer spacing,  $\theta$ -diffraction angle,

$$n = 1, 2, 3....$$

The grain size can be found out by using the Scherrer's formula (2-2) [56].

 $D = 0.9\lambda / \beta \cos\theta$  (2-2) Where:

- D is the mean size of the ordered (crystalline) domains, which may be smaller or equal to the grain size
- $\lambda$  is the X-ray wavelength;
- $\beta$  is the line broadening at half the maximum intensity (FWHM),
- $\theta$  is the Bragg angle.

## 2.10.2 Spacing of Lattice Planes

The perpendicular distance separating each lattice plane in a stack is denoted by the letter *d*, *d* spacing and the relationship to the particular lattice plane is  $d_{hkl}$  (i.e.,  $d_{001}$ ,  $d_{101}$ ,  $d_{103}$ ) with the Miller indices for the particular plane shown in the subscript, this is the common notation used in crystallography and X-ray diffraction. The values of d spacing in terms of hexagonal systems are shown in the equation (2-3), and for monoclinic systems are shown in equation (2-4) and for cubic systems are shown in equation (2-5) [57].

$$\mathbf{d} = \left[\frac{4}{3a^2}(\mathbf{h}^2 + \mathbf{h}\mathbf{k} + \mathbf{k}^2) + \frac{\mathbf{h}^2}{\mathbf{c}^2}\right]$$
 (2-3)

The values of d spacing for monoclinic is shown in the equation:

$$\mathbf{d} = \begin{bmatrix} \frac{\mathbf{h}^2}{\mathbf{a}^2 + \mathbf{c}^2} - \frac{2\mathbf{h}\mathbf{l}\cos\beta}{\mathbf{a}\mathbf{c}} \\ \frac{\mathbf{h}^2}{\mathbf{s}\mathbf{n}^2 \ \mathbf{\beta}} + \frac{\mathbf{h}^2}{\mathbf{b}^2} \end{bmatrix}$$
(2-4)

The value of d spacing for cubic is shown in the equation:

$$d = \sqrt[a]{h^2 + k^2 + l^2}$$
.....(2-5)

#### 2.11 Mechanical properties of ferrite

Mechanical properties are most important in ferrites. The ceramic nature of a ferrite makes it vulnerable to impact, thermal shock, and tensile failure. It is important for the ferrite designer to know the internal structure of this material, their mechanical workability, hardness, density [58].

### **2.11.1 Densities of ferrites**

Ferrites, being ceramic formed by sintering, have mechanical properties similar to those of pottery and in particular the properties depend on the sintered density. The pressed core before firing consists of relatively porous compact of oxides and during sintering the oxides react to form crystallites, or grains, of the required composition, the grains nucleate at discrete centers and grow outwards until the boundaries meet those of neighboring crystallites. During this process the density of the mass rises. Although there are some differences in X-ray densities owing to differences in divalent ions, the major contribution to effective density of ferrite part is its porosity [59]. The density of the sample can be measured by knowing the three dimensions of the sample, as shown in the equation below (2-6):

$$\rho = M(g) / V (cm^3)$$
 .....(2-6)

Where,  $\rho$ : density, M: mass of sample, V: volume of sample.

### 2.12 Diameter of grains

The Scherrer equation is used to determined the diameter of grains after smoothing and refining the sintered sample , This is done by enumeration the grains who the straight is extending across the image after enlarge, then it divides the length of the straight on the number of grains to find the diameter of grain rate. This test also helps to study the crystalline patterns emerging as well as the study of the form and distribution of crystals.

In most applications, data are collected over a selected area of the surface of the sample, and a 2-dimensional image is generated that displays spatial variations in these properties. Areas ranging from approximately 1 cm to 5 microns in width can be imaged in a scanning mode using that technique. The SEM is also capable of performing analyses of selected point locations on the sample; this approach is especially useful in qualitatively or semi-quantitatively determining chemical compositions [60].

## 2.14 Polarization

Electrical insulators have very few free electrons to take-part in normal electrical conductivity. Such a material has interesting electrical properties because of the ability of an electric field to polarize the material to create electrical dipole, thus insulating material moleculars are called (Non polar molecules) [61,62]. As well as appearing dipole in a material in the presence of a field, dipoles may be present as a permanent feature of the molecular structure. Such dipoles are called (Permanent dipoles) in which the center of the positive charge does not coincide with the center of the negative charges such insulating

material molecular are called (polar molecules). Induction of the dipoles is called electric polarization [63].

Phenomenon of polarization (P) down to the change in the arrangement of electrically charged particles of a dielectric in space, or is the surface charge density in a dielectric, equal to the dipole moment per unit volume of material being defined as follow:

$$P = Nm \qquad \dots (2-7)$$

where:

N: is the number of dipoles per unit volume.

m: is the average dipole moment.

The electric dipole moment corresponds to two electric charges of opposite polarity  $\pm q$  separated by the distance (d) [64]:

$$m = qd$$
 ...(2-8)

We can represent the electrical displacement (*D*) as the sum of the electric field (E) at a given point of dielectric and the polarization at the same point:

$$D = \mathcal{E}_{o}E + P \qquad \dots (2-9)$$

Where:

 $\varepsilon_{o}$ : is the permittivity of vacuum (8.85x10<sup>-12</sup> F/m)

The relationship between the electrical displacement and the electric field through a dielectric medium is:

$$D = \mathcal{E}_{o} \mathcal{E}_{r} \mathbf{E} \qquad \dots (2-10)$$

 $\epsilon_r$ : is called the relative permittivity or dielectric constant of the medium, for vacuum  $\epsilon_r$ =1, so

$$D = \varepsilon_{0} E \qquad \dots (2-11)$$

By substitute equation (2-10) in (2-9) we get [65, 66]:

$$P = \varepsilon_{o} \varepsilon_{r} \mathbf{E} - \varepsilon_{o} \mathbf{E}$$
$$P = \varepsilon_{o} (\varepsilon_{r} - 1) \mathbf{E} \qquad \dots (2 - 12)$$

### 2.15 Types of Polarization:

### 2.15.1 Electronic Polarization

Electronic polarization may be induced to one degree or another in all atoms. It results from a displacement of the center of the negatively charged electron cloud relative to the positive nucleus of an atom by the electric field. This polarization type is found in all dielectric materials and, of course, exists only while an electric field is present. as shown Figure (2-6a).

### 2.15.2 Ionic Polarization

Ionic polarization occurs only in materials that are ionic. An applied field acts to displace actions in one direction and anions in the opposite direction, which gives rise to a net dipole moment. As shown figure (2-6b).

### 2.15.3 Orientation Polarization

The third type, orientation polarization, is found only in substances that possess permanent dipole moments. Polarization results from a rotation of the permanent moments into the direction of the applied field. This alignment tendency is counteracted by the thermal vibrations of the atoms, such that polarization decreases with increasing temperature. as shown Figure (2-6c) [67].

# 2.15.4 Interfacial Polarization.

The final type of polarization is space-charge polarization, sometimes called interfacial polarization, and results from the accumulation of charge at structural interfaces in heterogeneous materials. Such polarization occurs when one of the phases has a much higher resistivity than the other, and it is found in a variety of ceramic materials, especially at elevated temperatures [68,69].

Other polarizations that based on the composition chemical of the material and its components and its called (Interfacial or space charge polarization), that occurs at frequencies of low little and very under audio waves, depending on the type of defects and heterogeneity that causes the polarization [70]. as shown Figure (2-6d).



Figure (2-6) Schematic diagram for the types of polarization.[68]

- a) Electronic Polarization
- b) Ionic Polarization
- c) Orientation Polarization
- d) d) Space charge Polarization

### 2.13 Electrical properties of ferrites

The electrical properties of ferrite substances play an important role in many electrotechnical applications. The factors that have the most influence on these properties are the purity of the constituent oxides and the effect of impurities on the final ferrite produced, the proportions and homogeneity in the powder mix and the control of temperature and atmosphere during sintering [57].

### 2.13.1 Dielectric constant

The permittivity means the charge storing capacity of a material. Consider two metal parallel plates of area (A) separated by a distance (d), in vacuum, attaching these plates to an electric circuit, the capacitance  $C_o$  of the parallel plates given by:

$$C_o = \frac{\varepsilon_o A}{d} \qquad \dots (2-13)$$

If a dielectric material is inserted between the capacitor plates, the capacitance (C) of the parallel plates will increase, as following:

$$C = \frac{\varepsilon A}{d} \qquad \dots (2-14)$$

Where:

 $\epsilon$ : is the of the dielectric material.

The relative permittivity, dielectric constant, of a material ( $\varepsilon_r$ ) defined as:

$$\varepsilon_r = \frac{\varepsilon}{\varepsilon_o} \qquad \dots (2-15)$$

Since  $\varepsilon$  is always greater than  $\varepsilon_o$ , the minimum value for  $\varepsilon_r$  is (1). By substitute equations. (2-15 in 2-14), the capacitance of the metal plates separated by the dielectric is

$$C = \varepsilon_r \frac{\varepsilon_o A}{l} \qquad \dots (2-16)$$

$$C = \varepsilon_r C_0 \qquad \dots (2-17)$$

Thus  $\varepsilon_r$  is a dimensionless parameter that compares the charge-storing capacity of a material to that of vacuum. Dielectric constant depends upon the frequency of the applied electric field. It decreases with the increase in frequency. Dielectric constant also depends upon temperature [67, 71, 72].

### 2.13.2 Electrical resistivity

Ferrites is a semi-conductor material has a good electrical resistance, the state of parity between ferrous and ferric ionic is exchange where the excess

electron in the ferrous ion obtaining a little energy it's can move to a similar site near the ferric ion, and thus the excess electrons can jump between this ions under the influence of an electric field of applied. And that the resistivity decreases with increasing temperature this is because of it is semiconductor materials [60].

We apply this relationship (2-18) to measure the electrical resistivity.

$$\boldsymbol{\rho} = \boldsymbol{R} \frac{\boldsymbol{A}}{\boldsymbol{\ell}} \qquad \dots (2-18)$$

Where (R) is electrical resistance, (A) represents the surface area, and  $(\ell)$  is the thickness of the model. The resistivity depends on temperature as in the relationship (2-19) [41].

$$\boldsymbol{\rho} = \boldsymbol{\rho}_{o} \exp(\mathbf{E}/\mathbf{kT}) \qquad \dots (2-19)$$

where E activation energy, T temperature, k is the Boltzmann constant (k =8.62 \*10<sup>-5</sup> ev .k<sup>-1</sup>) and ( $\rho_0$  is a constant).

### 2.13.3 Dielectric loss

When an electric fields acts on any matter the latter dissipates a certain quantity of electric energy that transforms into heat energy. This is known as "loss" of power, i.e., the dissipation of an average electric power in matter during a certain interval of time. If a metal conductor is first connected to direct voltage and then to alternating voltage, the acting magnitude of which is equal to direct voltage, the loss of power ( $P_w$  in watt) in the conductor will be the same in both cases in conformity with the Joul-lenze law and equal to [71,73]:

$$P_w = V^2/R$$
 ...(2-20)

Where V is the voltage in volt and R is the resistance of the conductor in ohms. As distinct from conductors, most of the dielectrics display a characteristic feature, under a given voltage the dissipation of power in these dielectrics depends on the voltage frequency.

The expense of power at an alternating voltage is markedly higher than at a direct voltage, and rapidly grows with an increase in frequency, voltage, and capacitance, and also depends on the materials of the dielectric.

A dielectric loss is an amount of power loss in an electrical insulator. Dielectric losses at a direct voltage can easily be found from eqn. (2-20) where R stands for the resistance of the insulator, while the losses under an alternating voltage are determined by more intricate regularities. When considering dielectric losses we usually mean the losses precisely under an alternating voltage. Let the alternating voltage (V=V<sub>o</sub>exp (j $\omega$ t)) be applied to a circuit containing a capacitor, with air as a dielectric medium. The current (I) passing through the capacitor according to Ohm's law.

$$I = \frac{V}{X_c} \qquad \dots (2-21)$$

Where X<sub>c</sub> is the impedance of the capacitor of a capacitance C

$$X_c = \frac{1}{\text{Cj}\omega} \qquad \dots (2-22)$$

Considering the dielectric material in a capacitor (C),  $\omega=2\pi f$ , where,  $\omega$  is the angular frequency f is the frequency

$$j = \sqrt{-1}$$

The current I may be calculated by substitute equation (2-17) in (2-22) and the result in equation (2-21) as follow,

$$I = j\omega\varepsilon_{\rm r}C_{\rm o}V \qquad \dots (2-23)$$

The current I in a dielectric is  $90^{\circ}$  advanced in phase in relation to the voltage as shown in Figure (2-7), this implies that we have a capacitive component of the current, then the heat for the system equal to zero, thus the energy is transferred in a dielectric without losses and emission of heat "Ideal dielectric".





In actual fact the phase angle  $\varphi$  is slightly less than 90°, the total current I through the capacitor can be resolved into two components active (resistive current) I<sub>a</sub> and reactive (capacitive current) I<sub>r</sub> currents [74].

The phase angle is very close to  $90^{\circ}$  in a capacitor with a high quality dielectric, the angle  $\delta$  is a more descriptive parameter which, when added to the angle  $\varphi$ , brings the angle  $\varphi$  to  $90^{\circ}$ .

$$\delta = 90^{\circ} - \varphi \qquad \dots (2 - 24)$$

The angle  $\delta$  is the dielectric loss angle. If  $\delta$  is small, therefore  $\sin \delta \approx \tan \delta$ . The tangent of this angle is equal to the ratio between the active and reactive currents:

$$\tan \delta = \frac{I_a}{I_r} \qquad \dots (2-25)$$

 $\tan \delta$  is the dielectric loss tangent or dissipation factor. It is important to note that the dielectric response of a solid can be succinctly described by expressing the relative dielectric constant as a complex quantity,

$$\varepsilon_r = \varepsilon_r' - j\varepsilon_r'' \qquad \dots (2-26)$$

In which  $\varepsilon'_r$  is the real component of dielectric constant of the material, and  $\varepsilon''_r$  is the imaginary component, is known as the dielectric loss factor [75, 76].

We can find the value of  $I_a$  and  $I_r$  by substituting equ. (2-26) in equ. (2-23):

$$I = j\omega(\varepsilon'_r - j\varepsilon''_r)C_oV \qquad \dots (2-27)$$
$$I = \omega\varepsilon''_r C_oV + j\omega\varepsilon'_rC_oV \qquad \dots (2-28)$$

$$I = \omega \varepsilon_r'' C_o V + j \omega \varepsilon_r' C_o V \qquad \dots (2-28)$$

The total current (I) in terms of the components  $I_a$  and  $I_r$  are:

$$I = I_a + jI_r \qquad \dots (2-29)$$

$$I_a = \omega \varepsilon_{\rm r}^{\prime\prime} C_{\rm o} V \qquad \dots (2-30)$$

$$I_r = \omega \varepsilon'_r C_o V \qquad \dots (2-31)$$

By substituting equations (2-30) and (2-31) in eqn. (2-25) we can get

$$\tan \delta = \frac{\varepsilon_r''}{\varepsilon_r'} \qquad \dots (2-32)$$

The quality factor (Q) of an insulator portion is determined, the reciprocal value of the loss tangent [77]:

$$Q = 1/\tan\delta = \cot\delta \qquad \dots (2-33)$$

The expression form for the value of dielectric losses  $P_w$  in an insulation portion having a capacitance C [78]:

$$\mathbf{P}_{\mathbf{w}} = VI_a = VI_r \tan \delta \qquad \dots (2-34)$$

## 2.14 Applications of the hard ferrites

Owing to its low price, ferrite magnets are used for a number of applications ranging from magnetic holding tools to motors and generators. The ferrites have replaced other magnet materials in the existing systems, either with or without modification to the system. This is particularly the case with static applications where small demagnetizing fields are involved. A typical example is the application of flat ring instead of high metallic center core magnets in loudspeaker systems. The high H<sub>c</sub> has stimulated the development of the new system, especially in the dynamic application where periodically high demagnetizing fields are present. A typical example is the electric motor with its strong armature electric field.

New electric motors are being developed which require very high  $H_{cj}$  value lying far beyond the range of Al-Ni-Co materials, for example the starter motor, requiring  $H_{cj} > 320$  kA/m.

Applications of hard ferrite other than permanent magnets are in the field of microwave, magnetic bubble memories, magnetic tape recording and magneto-optics. M-type ferrites are of interest for resonance type microwave devices e.g. isolators, filters, and circulators. Below 20 GHz, such devices normally employ a garnet or spinel ferrite in combination with the bias magnets. At high frequencies, the required bias field becomes impracticably high (>570 kA/m). M-type ferrites are then preferred because of their large anisotropic field which acts as built-in bias field and provide a resonance frequency of about 50 GHz in a small tuning field.

For broader frequency coverage, various substitutions can be employed by the alter of HA. Other, application of magnets in magneto-therapy, purification, magnetic bearing, and automatic camera are a few more applications, which are increasingly becoming important. Some of the common applications in device used in everyday life are highlighted in table (2-3) [42]:

Devices	Parts
Cassette recorder	Speaker, synchronous motor, mike etc
Video cassette recorder	Main wheel motor
TV sets	Speaker, color adjusting magnet
Air conditioner	Fan motor
Refrigerator	Fan motor, compressor motor, rubber
	lining
Car	Starter motor, window motor, viper motor
Computers	Disk drive, fan motor, speaker etc
VCD and DVD	Main wheel motor

 Table (2-3) Common application of BaM ferrites



# **Experimental Part**

# **3.1 Introduction**

In this chapter the details of sample preparation as including, weighing, mixing, precipitation, filtering and various characteristic techniques used to produce barium ferrite and inherent parameters are explained.

# 3.2 The materials used

The chemical materials used for preparing barium ferrite are shown in table (3-1) together with their properties:

Compounds	Chemical formula	% Purity	Origin
FeCl <sub>3</sub> .6H <sub>2</sub> O	Ferric chloride	≥99%	India
BaCl <sub>2</sub> .2H <sub>2</sub> O	Barium chloride	≥99%	India
NaOH	Sodium hydroxide	≥99%	India
C <sub>2</sub> H <sub>5</sub> OH	Ethanol	33	Riedal

 Table (3-1): The chemical materials and their properties

# 3.3 Method of preparation of barium ferrite in aqueous solvent

FeCl<sub>3</sub>.6H<sub>2</sub>O and BaCl<sub>2</sub>.2H<sub>2</sub>O with a Fe/ Ba molar ratio of (15) are dissolved in water. The molar ratio of Fe /Ba is 15 used to maintain the stoichiometry of the product due to the poor solubility of  $Ba^{+2}$  cations in water. The prepared solutions are co-precipitated by adding of NaOH with OH/Cl<sup>-</sup> molar ratio 2 at room temperature.

 $65.67 \text{ gm of FeCl}_3.6H_2O$  is dissolved in 150 ml of distilled water in beaker (a<sub>1</sub>), 2.193 gm of BaCl<sub>2</sub>.2H<sub>2</sub>O is dissolved in 40 ml of water in another beaker (a<sub>2</sub>), 65 gm NaOH is also dissolved in 600 ml of distilled water in third beaker (a<sub>3</sub>). (a<sub>1</sub>) is mixed with (a<sub>2</sub>) gradually, and then (a<sub>3</sub>) is mixed with them also gradually, then it is noticed that the material be deposited. The sample synthesized in distilled water solutions is washed by distilling water, filtering, and drying at 80 °C for 12 h. Then, the dried powder has been calcined at 800 and 1000°C for 2 h in air to obtain barium ferrite. The schematic representation of the main steps employed in the preparation shown in figure (3-1):



Figure (3-1) Preparation steps of barium ferrite nano powders in aqueous solution



Figure (3-2) Diagram show preparation of barium ferrite in aqueous solution

# **3.4 Method of the preparation of barium ferrite using ethanol/water solvent**

FeCl<sub>3</sub>.6H<sub>2</sub>O and BaCl<sub>2</sub>.2H<sub>2</sub>O with a Fe/ Ba molar ratio of 15 are dissolved in ethanol/water with a volume ratio of 3:1, 65.67 gm FeCl<sub>3</sub>.6H<sub>2</sub>O, is dissolved in 100 ml of ethanol/water solution in beaker (b<sub>1</sub>), 2.193 gm of BaCl<sub>2</sub>.2H<sub>2</sub>O is dissolved in 50 ml of ethanol/water in another beaker (b<sub>2</sub>). Then in third beaker (b<sub>3</sub>), 65 gm of NaOH was dissolved in 400 ml of ethanol/water solution, (b<sub>1</sub>) is mixed with (b<sub>2</sub>) gradually, and then (b<sub>3</sub>) is added to them so gradually, the sample synthesized is washed by ethanol / water solution with a volume ratio 2:1, filtered, and dried at 80 °C for 12 h. Then, the dried powder is calcined at 800 and 1000 °C for 2 h in air to obtain barium ferrite. The schematic representation of the main steps employed in the preparation is shown in figure (3-3):



Figure (3-3) Preparation steps of barium ferrite nano powders using ethanol/water solution



Figures (3-4): Preparation of barium ferrite Using 3:1 ethanol/water solution

# 3.5 Equipment (mold)

There are many types of equipment used in powder compacting. These are the molds. A mold is designed for the manufacture of samples in the form of pellet in diameter (2cm) and thickness (4mm) and the weight of the sample is (3 g). It uses hydraulic press with a pressure of 500-700 psi. Figure (3-5) illustrates the mechanism of powder consolidation



Figure (3-5) Mechanism of powder consolidation [79].

# **3.6 Forming**

Since ferrite parts are formed by pressing. The granules are poured in to a suitable die and then compressed. It is still very fragile and requires sintering to obtain the final ferrite properties. It is preferable to have a powder of various sizes of particles which increases the durability and fills in the spaces between the big Particles. Figure (3-6) shows the transformation of powder to compact sample.



Figure (3-6) Transformation of powder to compact sample

# **3-7 Sintering**

Sintering may be defined as the heating of loose or compacted aggregate of metal powder below the melting point of the base metal with or without the application of external pressure in order to transform it to a more dense material by interparticle bonding. Sintering process is concerned with the diffusion of particle to particle , the formation of grain boundary and the closing of voids present in the green briquettes. Sintering is a rather very complex process in which physical, chemical and metallurgical effects interact because of the numerous changes occurring simultaneously within the material. It is very difficult to define and analyse sintering under heat; bonding takes place between the porous aggregate particles and once cooled powder has also bonded to form a solid piece. Sintering can be considered to proceed in three stages:

- 1- Neck growth proceeds rapidly but powder particles remain discrete.
- 2- Most densification occurs, the structure recrystallizes and particles diffuse into each other.
- 3- Isolated pores tend to become spheroidal and densification continues at a much lower rate. Figure (3-7) shows compact powder before and after sintering:



**Before Sintering** 

After Sintering

Figure (3-7) Powder metal compact before and after sintering [80].

# 3.8 Instruments

We have used a set of devices for the purpose of testing the prepared samples.

# 1. Balance

We have used sensitive balance with high degree of sensitivity of four digits, Type "GÖTTINGEN ", Max. 210 gm, Germany origin .

# 2. Magnetic Stirrer

It is a laboratory device that employs a rotating magnetic field to cause a stir bar immersed in a liquid to spin very quickly, thus it is stirring it. The rotating field may be created either by a rotating magnet or a set of stationary electromagnets, placed beneath the vessel with the liquid. Magnetic stirrers often include a hot plate or some other means for heating the liquid, for obtained homogenous mixer between atoms and liquid particles, Type " Wisestir ", work at 20 A and 50/60 Hz, made by "DAIHAN" Scientific Co. Ltd., Korea origin.

# 3. Electrical furnace

The prepared three nanopowders are calcined in an electrical furnace with size chamber of 40x20x20 cm. The thermal regime of the furnace is controlled

through "Eurotherm" programmer-cum-controller, Type-KR 170 E , Max temp. 1150  $^\circ C$  , 220 V , 13.8 A , 50/60 Hz , Japan origin .

# 4. X-Ray Diffractometer

Powder type X-ray Diffractometer (XRD-6000, Shimadzu, Japanese origin) which uses  $CuK_{\alpha}$  (1.54060A°) as a radiation source operated at 40 kV and 30 mA is used for the verification of crystal structure and the average crystallite size of particles. The data are collected in the 2 $\theta$ range from 20° to 65° with a step of 0.04° and counting time of 1sec/step.

# 5. Scanning electron microscope (SEM)

The scanning electron microscope used in imaging the nanoparticles is a VEGA//Easy Probe which is a favorable combination of a scanning electron microscope and a fully integrated energy dispersive X-ray microanalyses , (magnification ranging from 20X to approximately 30, 000X, spatial resolution of 50 to 100 nm), produced by TESCAN, s.r.o., Libušina trída 21.

### 6. LCR meter

The type of LCR meter is Agilent impedance analyzer an American origin, its range of frequency is (50Hz-5MHz), as shown in the figure (3-8).



Figure (3-8) : LCR meter used in electrical measurement

# **3.9 Electric Tests**

# **3.9.1 Dielectric Constant**

The dielectric constant which is measured by LCR meter connected to computer that the sample which puts between the poles and makes sure that the poles touch sample surface [60].

LCR system is used for the purpose of measuring the capacity of the disc samples at different frequencies. Dielectric constant data have recorded on computer's screen via mathematic formulas that studied previously.

### **3.9.2 Dispersion Factor (Tangent Loss)**

The Tangent Loss was measured by LCR meter which connected to computer the sample puts between the poles and make sure that the poles touch sample surface. Dispersion factor data have recorded on computer's screen via mathematic formulas that studied previously.

### **3.9.3 Electrical resistivity**

This is the electrical resistance of a ferrite core, having a constant crosssectional area and its unit is ohm-cm. In the present work, the surface of the pellets is cleaned by grinding with SiC paper in order to remove any contamination and then used to study the room temperature resistivity; silver paste is used to coat the polished pellets to provide electrical contacts. AC -resistivity is measured by a two-probe method using LCR meter.



# Results and Discussion

## **4.1 Introduction**

This chapter includes the results and discussion of the physical testing and the characterization (X-ray analysis, Scanning Electron Microscope (SEM), Density, Hardness, Dielectric constant, Electrical resistivity, Tangent loss and Electrical conductivity).

# **4.2 X-ray Diffraction analysis**

XRD can be used to characterize the crystallinity of nanoparticles, and the average diameters of all the nanoparticles. The precipitated fine particles are characterized by XRD for structural determination and estimation of crystallite size. The lattice constant (a) is computed using the 'd' value with their respective (h k l) parameters. The ferrites prepared in the present work are phase analyzed by comparison with standard XRD.

# **4.2.1 XRD** pattern of the barium ferrite prepared using aqueous solutions

This powder is prepared at room temperature and calcined at 800 °C and 1000 °C for 2 hours.



Figure (4.1 a): XRD pattern of barium ferrite prepared in aqueous solution at room temperature



Figure (4.1 b): XRD pattern of barium ferrite prepared in aqueous solution and calcined at 800 °C



Figure (4.1 c): XRD pattern of barium ferrite prepared in aqueous solution and calcined at 1000 °C

Figures (4.1: a, b and c) show the XRD pattern of barium ferrite prepared using aqueous solutions at room temperature .The appearance of some phases

shows the part of the oxides of raw materials at  $2\theta$ =(36.62,21.23) because the barium ferrite phase isn't growth yet like (Fe<sub>2</sub>O<sub>3</sub>), at  $2\theta$ =31.70 which there is a partial phase grown of barium ferrite.

Then, the growth of barium ferrite phase causes the low values of magnetic permeability which lead to emergence of poor electrical properties .

Figure (4.1 b) at 800 °C, there is slow growing of the barium ferrites phases because the temperature is still low, but some peaks described in figure (4.1 a) are disappeared in figure (4.1 b) like those at  $2\theta$ = (45.46).

Figure (4.1 c) XRD pattern at 1000 °C shows fully the growth of crystalline barium ferrites phases. When comparing the charts with the ASTM cards shown in the appendix, it can be noticed the emergence of most of the peaks of barium ferrite. The detailed analysis of the XRD and the assignments of various reflections are given in the Tables (4.1 a), (4.1 b):

 Table (4.1 a): Strongest three peaks of ferrite prepared in aqueous solution and calcined (at room temperature)

N.	20		FWHM	Intensity	1-1-1	Card
NO.	(degree)	d(A)	(degree)	I/I <sub>1</sub>	NKI	NO.
1	31.7050	2.81994	0.23000	100	(110)	200127
2	36.6200	2.45195	0.42660	76	(311)	261136
3	21.2300	4.18167	0.58000	47		210920

Table (4.1 b): Strongest three peaks of ferrite prepared in aqueous solution and calcined (at 800 °C)

NO	20	J(Å)	FWHM	Intensity	1-1-1	Card
NU.	(degree)	d(A)	(degree)	I/I <sub>1</sub>	hKl	NO.
1	33.2291	2.69400	0.18960	100	(104)	011053
2	35.6939	2.51342	0.16670	60		280142
3	54.1387	1.69271	0.15020	55	(422)	021047

calchieu (at 1000 °C)								
NO	20	d(Å)	FWHM	Intensity	hl/l	Card		
NO.	(degree)	u(A)	(degree)	$I/I_1$	IIKI	NO.		
1	29.7850	2.99720	0.11890	100	(130)	011053		
2	33.4476	2.67690	0.12850	53	(200)	280142		
3	54.3354	1.68705	0.11800	24	(211)	021047		

Table (4.1 c): Strongest three peaks of ferrite prepared in aqueous solution and calcined (at 1000 °C)

# Grain size calculation from X-ray diffraction for barium ferrite

From XRD, average grain size has been estimated using Debye-Scherrer formula (2-2):

The calculated particle size for sample calcined at 800  $^\circ\text{C}$  :

D =43.7 nm and for sample calcined at 1000  $^{\circ}$ C :

D =69.1 nm

# 4.2.2 XRD pattern of the barium ferrite prepared using ethanol/water solution

Here the powder is prepared at the same temperatures like the previous method

(4.2.1) The XRD pattern of the synthesized barium ferrite powder are shown in Figure (4-2: a, b, and c).



Figure (4-2 a): XRD pattern of the barium ferrite prepared in ethanol/water solution at room temperature



Figure (4-2 b): XRD pattern of the barium ferrite prepared in ethanol/water solution and calcined at 800  $^\circ\mathrm{C}$ 



Figure (4-2 c): XRD pattern of the barium ferrite prepared in ethanol/water solution and calcined at 1000 °C

Figures (4-2: a, b and c) show the XRD for the barium ferrite prepared using ethanol/water as a solvent at different temperature. All diffraction data are in good agreement with ASTM cards shown in appendix.

In figure (4.2 a) at room temperature, barium ferrite phases in addition to iron oxide appears.

Figure (4.2 b) at 800 °C and (4.2 c) at 1000 °C shows full growth of barium ferrites phases by comparing the charts with the ASTM cards shown in the appendix, it can be noticed the emergence of most of the peaks of barium ferrite, at figure (4.2 c) it is observed that three strongest sharp peaks which mean that FWHM is very small so crystalline increases as the calculation of the particle size is prove.

The detailed analysis of the XRD and the assignments of various reflections are given in tables (4-2 a, 4-2 b):

NO	20	۲ (Å)	FWHM	Intensity	1-1-1	NO.
NO.	(degree)	d(A)	(degree)	$I/I_1$	nki	Card
1	31.7989	2.81183	0.18740	100	(110)	200127
2	45.5105	1.99149	0.16000	52	(200)	200127
3	45.7303	1.98243	0.10000	15	(722)	290172

 Table (4-2a) Strongest three peaks of ferrite prepared in ethanol/water solution and calcined (at room temperature)

Table (4-2a) Strongest three peaks of ferrite prepared in ethanol/water solution and calcined (at 800 °C)

	20		FWHM	Intensity	111	NO.
NO.	(degree)	d(A)	(degree)	$I/I_1$	hKI	Card
1	31.7775	2.81367	0.16990	100	(110)	200127
2	45.5109	1.99148	0.15200	48	(200)	200127
3	34.1412	2.62409	0.19500	31	(441)	290172

Table (4-2 b) Strongest three peaks of ferrite prepared in ethanol/water solution and calcined (at 1000 °C)

NO.	20	d(Å)	FWHM	Intensity	hkl	NO.
	(degree)		(degree)	$I/I_1$		Card
1	34.3009	2.61223	0.22190	100	(114)	430002
2	32.4098	2.76021	0.12140	69		280144
3	29.7811	2.99759	0.09500	54		280144

# Particle size calculation from X-ray diffraction of (Ba Fe<sub>11</sub>O<sub>19</sub>)

# (ethanol / water as a solvent)

The calculated particle size for sample calcined at 800 °C:

D = 35.8 nmand for sample calcined at 1000 °C :

D = 48.6 nm.
#### 4.3 Scanning electron microscope (SEM) test

SEM micrograph of  $(BaFe_{12}O_{19})$  pellet shows the structural of the sample (Barium ferrite prepared using aqueous solution at 1000 °C), (Barium ferrite prepared using ethanol/water as a solvent at 1000 °C). The grains are more or less spherical in shape and there is grain size distribution over different samples prepared by the ceramic method. The rate of grain growth and the resultant microstructure depends on a complex way of many factors, such as the sintering temperature, preparation method and stoichiometry. The images were obtained are similar to images who obtained by the researcher Q.Mohsen [24].

The SEM images of samples are shown in figures (4-3: a, b, c, d, e) and (4-4: a, b, c, d, e).



Figure (4-3 a): SEM image of barium ferrite prepared in aqueous solution and calcined at 1000 °C



Figure (4-3 b) SEM image of barium ferrite prepared in aqueous solution and calcined at 1000 °C



Figure (4-3 c): SEM image of barium ferrite prepared in aqueous solution and calcined at 1000  $^\circ\mathrm{C}$ 



Figure (4-3 d): SEM image of barium ferrite prepared in aqueous solution and calcined at 1000  $^\circ\mathrm{C}$ 



Figure (4-3 e): SEM image barium ferrite prepared in aqueous solution and calcined at 1000  $^\circ\mathrm{C}$ 



Figure (4-4 a): SEM image of barium ferrite prepared in ethanol/water solution and calcined at 1000 °C



Figure (4-4 b): SEM image of barium ferrite prepared in ethanol/water solution and calcined at 1000 °C



Figure (4-4 c): SEM image of barium ferrite prepared in ethanol/water solution and

#### calcined at 1000 °C



Figure (4-4 d): SEM image of barium ferrite prepared in ethanol/water solution and calcined at 1000 °C



Figure (4-4 e): SEM image of barium ferrite prepared in ethanol/water solution and calcined at 1000 °C

Scanning electron microscope (SEM) for the samples sintered at 1000°C is used to determine the lump size. It seems that the particles morphology is affected by the type of solvent.

From the SEM images, it is observed that the formation of soft agglomerates with irregular morphology constituting the quite fine particles. The study of SEM micrographs reveals a less number of pores with smaller lump size, and a homogeneous system with agglomerates of submicron particles. Since the ceramic method involves sintering of the stoichiometric mixtures at high temperatures, the crystallites are in order of micrometer with relatively smooth surface. The aggregate of crystallites of various sizes indicates a size distribution in the micrographs. The variation in size of the particles among the different samples is because of undistributed temperature equilibrium through the sample. When the ferrite powders with more crystalline content are used for samples sintered at high temperatures, the local shrinkage speed is much higher than the densification speed of the ceramics and also it shows a homogeneous system with agglomerates of submicron particles. SEM images of barium ferrite also reveal a great decrease of particle size, and a loss of the original morphology of the particles. It also reveals a decrease in calcinations temperature resulting to particles being finer and more uniform as we observe in figure (4-3: a, b, c, d and e), (4-4: a, b, c, d and e).

#### 4.4. Electrical measurements

In order to indicate the extent of appropriate ferrite for the practical application, some important electrical tests were conducted which includes measuring electrical resistivity of the samples as well as the dielectric constant, and electrical loss tangent by using LCR meter at range of frequencies [50 Hz-5MHz]. In order to find the best ferrite this maintains high values for electrical insulation and the electric loss tangent a low within the frequency specific at which being the measurement.

# 4.4.1 Measurements of electrical resistivity (ρ) and electrical conductivity (σ)

Electrical resistivity ( $\rho$ ) was measured for samples which were prepared at different degrees of calcined temperature. There are some factors which important to determining the resistivity of ferrites there are grain size, grain boundaries, porosity and stoichiometric. That is why samples sintered at lower temperatures which have smaller grain sizes exhibit higher values of resistivity, that resistivity of a polycrystalline material, in general, increases with the decreasing grain size. Smaller grains imply larger number of insulating grain boundaries which act as barriers to the flow of electrons. Smaller grains also imply smaller grain-to-grain surface contact area and therefore a reduced electron flow. The following figures (4-5), (4-6), (4-7) and (4-8) show the resistivity as a function of frequency for all samples:



Figure (4-5): Resistivity as a function of frequency of barium ferrite prepared in aqueous solution and calcined at 800 °C



Figure (4-6): Resistivity as a function of frequency of barium ferrite prepared in aqueous solution and calcined at 1000 °C



Figure (4-7): Resistivity as a function of frequency of barium ferrite prepared using ethanol/water solution as a solvent and calcined at 800 °C



Figure (4-8): Resistivity as a function of frequency of barium ferrite prepared in ethanol/water solution and calcined at 1000 °C

The previous figures showed that electrical resistivity values decrease when the frequency increased. It also showed that the resistivity of the ferrites decreases with increasing of the sintering temperature The formation of  $Fe^{+2}$ gives rise to electron hopping between  $Fe^{+2}$  and  $Fe^{+3}$  ions which bring about a reduction in resistivity [81]. High resistivity ferrites with low eddy current losses are required from the technological applications point of view. The electrical resistivity of ferrites has been normally found to increase on doping. The electrical resistivity is increased due to the increasing of the separation between the magnetic ions whereas the number of charge carriers remains unchanged. For information the ferrite phase growth is uncompleted at low temperatures which increase the resistivity because of the existing of some oxides that doesn't interact, then after the increasing of the temperature the ferrite phase growth happens which causes decreasing of the resistivity.

Resistivity of ferrites is known to depend upon the purity of the starting materials and the preparation details such as sintering temperature. Then the conductivity is inverted resistivity, and it increases whenever decreased resistivity.



Figure (4-9) Conductivity as a function of frequency of barium ferrite prepared in aqueous solution and calcined at 800 °C



Figure (4-10): Conductivity as a function of frequency of barium ferrite prepared in aqueous solution and calcined at 1000 °C



Figure (4-11): Conductivity as a function of frequency of barium ferrite prepared in ethanol/water solution and calcined at 800 °C



Figure (4-12): Conductivity as a function of frequency of barium ferrite prepared in ethanol/water solution and calcined at 1000 °C

The conductivity is also dependent upon the purity of the starting materials, the preparation details such as sintering temperature and the influential frequency that's mean when the frequency increased conductivity increase; figures (4-9), (4-10), (4-11) and (4-12) shows this dependence.

# 4.4.2 Measurements of Dielectric constant ( $\mathcal{E}_r$ ) and electrical loss tangent (tan $\delta$ )

The dielectric constant  $(\varepsilon_r)$  calculated using equation (2-10), shows a change in the dielectric constant as a function of applied frequency. Absence of the ions movement, polarization orientation and the decrease in dielectric constant mainly cause the movement of polar groups under the influence of an electric field , Figures (4-12),(4-13),(4-14),(4-15) show the dielectric constant as a function of frequency .



Figure (4-13): Dielectric constant as a function of frequency of barium ferrite prepared in aqueous solution and calcined at 800 °C



Figure (4-14): Dielectric constant as a function of frequency of barium ferrite prepared in aqueous solution and calcined at 1000 °C



Figure (4-15): Dielectric constant as a function of frequency of barium ferrite prepared in ethanol/water solution and calcined at 800 °C



Figure (4-16): Dielectric constant as a function of frequency of barium ferrite prepared in ethanol/water and calcined at 1000 °C

A high value of  $(\varepsilon_r)$  is observed at lower frequencies which later falls down rapidly with frequency increase. This behavior is typical of ferrites and a similar behavior was observed by several authors [62]. The trend can be explained on the basis that at lower frequencies there is four different types of polarization exist (i.e. electronic, ionic, dipolar and space charge) contributions take a part in the dielectric constant, but at higher frequencies some of polarization contributions relax out, result in the lowering of dielectric constant  $(\varepsilon_r)$ . The frequency of electron hopping between the Fe<sup>+2</sup> and Fe<sup>+3</sup> ions at octahedral sites is higher as compared to the applied AC field and thus can interact with the applied field easily, resulting in a higher value of dielectric constant at lower frequencies. Contrary to it, at higher frequency the hopping electron cannot follow the frequency of the applied electric field, resulting in lowering of dielectric constant. Consequently, the electron exchange between Fe<sup>+2</sup> and Fe<sup>+3</sup> is perturbed at high frequencies, which explains the slower decrease of dielectric constant ( $\varepsilon_r$ ) at high frequency.

It is observed that the influence of the pores shrinking that cause density increase and better homogeneity of the mixture on the dielectric constant of the prepared ferrite which increases at low values of frequencies and then start sliding downward within the frequency range (50Hz – 5MHz).

The different values of the constants of the insulating material leads to the possession of a different resonant frequency, at a certain frequency the movement of electric dipole become in the case of resonance then the ferrite block absorbing ability of the influential electric field, but at high frequencies, it cannot be polar electric diodes follow the effective electric field leads to a decline in the values of the dielectric constant of the material.

The values of the dielectric constant are within the range (40) at the low frequency to reach (0.5 F/m) at highest frequency of measurement was conducted.

The values of loss tangent of dielectric constant (tan  $\delta$ ) it has ranged between (2 – 0.1) at frequencies within the range (50Hz – 5MHz) respectively, Suddenly decreasing after this level to low values with increasing frequency.

The values of magnetic loss tangent represents the ratio between the dissipated energy to stored energy, previous figures show that the values of loss tangent is high at low frequency (50Hz-1KHz), then begin to decline at the frequency (1KHz-1MHz).

This values are varies from one compound to another depending on the type of ferrite mixture, and so it's less of prepared ferrite at high degrees of sintering, the high values of the magnetic loss tangent at sintering temperature 800°C it result of the presence of pores and small grain size, and possibly micro cracks that works to stop the movement of the wall of the magnetic field so leads these things combined to decreasing the magnetic permeability [62].



Figure (4-17): The variation of loss tangent with frequency of barium ferrite prepared in aqueous solution and calcined at 800 °C



Figure (4-18): The variation of loss tangent with frequency of barium ferrite prepared in aqueous solution and calcined at 1000 °C



Figure (4-19): The variation of loss tangent with frequency of barium ferrite prepared in ethanol/water solution and calcined at 800 °C



### Figure (4-20): The variation of loss tangent with frequency of Barium ferrite prepared in ethanol/water solution in and calcined at 1000 °C

## 4.5 Density test

Experimentally, density measurements are performed by knowing the dimensions of the model; we can find the density through measuring the dimensions of the samples, according to the following equation:

 $d=W(g)/V(cm^3)$  .....(4-1) where, W: is the weight of sample , V : is the volume of sample, the bulk

density is shown in table (4-4 a, b):

Table (4-3 a): The values of bulk density of barium ferrite prepared in aqueous solution

Sample	Density (d) g/cm <sup>3</sup>
Barium ferrite prepared in aqueous solution and calcined at 800 °C	2.19339
Barium ferrite prepared in aqueous solution and calcined at 1000 °C	2.59583

 Table (4-3 b): The values of bulk density of barium ferrite prepared in ethanol/water solution

Sample	Density (d) g /cm <sup>3</sup>
Barium ferrite prepared in ethanol/water solution and calcined at 800 °C	2.93593
Barium ferrite prepared in ethanol/water solution and calcined at 1000 °C	3.13122

There is an increase in the density and shrinkage of the sintered pellets with an increase of time, temperature and preparation method, this table confirms that the density properties increase with the increase of the sintering time and temperature.



# Conclusions and Recommendations

#### **5.1 Conclusions**

The chemical method used is more economic than the ceramic one because the ferrite can be prepared at 300 °C lower and this will results in energy saving.

## **5.2 Recommendations**

Based on the findings of this study, the following recommendations are put forward:

1. Using other preparation routes such as precipitation, co-precipitation, solgel, milling, and other.

2. Using a different ratio of salts in order to see there effect on the particle size, electrical properties of the ferrites like absorbance of the ferrite.

3. Using high temperature sintering more than it has been used in the current research.

4. Studying the magnetic properties (hysteresis loop) in order to get a new feature and advantage of it in the modern scientific applications.



#### **References:**

[1] S.P.Gubin, Y.u. Koksharov, G. B. Khomutov, G. Y. Yurkov, "Magnetic nanoparticles: preparation, structure and properties", Russian Chemical Reviews 74 (6), P. 489 – 520, (2005).

[2] ] I.Chicinas, "Soft magnetic nanocrystalline powders produced by mechanical alloying routes", Journal of optoelectronics and advanced materials, V. 8, 2, P. 439 – 448,(2006).

[3] William H, Von Aulock, "Hand Book of Microwave Ferrite Materials", a cademic Press, New York, London, (1965).

[4] B.H. Sohn, R.E. Cohen, "Processible optically transparent block copolymer films containing superparamagnetic iron oxide nanoclusters", Chem. Mater. 9, P.264–269, (1997).

[5] C. Caizer, M. Popovici and C. Savii, "Spherical (ZndNi1-dFe2O4) nanoparticles in an amorphous (SiO<sub>2</sub>)1- $\gamma\gamma$  matrix, prepared with the sol-gel method", Acta Mater. 51, P. 3607–3616, (2003).

[6] D.K. Kim, Y. Zhang, W. Voit, K.V. Rao and M. Muhammed, "Synthesis and characterization of surfactant-coated superparamagnetic monodispersed iron oxide nanoparticles", J. Magn. Mater. 225, P. 30–36, (2001).

[7] C. Liu, B. Zou, A.J. Rondinone, Z.J. Zhang, "Reverse micelle synthesis and characterization of superparamagnetic MnFe<sub>2</sub>O spinel ferrite nanocrystallites", J. Phys. Chem. B 104, P. 1141–1145, (2000).

[8] H. Gleiter, Prog. Mater. Sci. 33, P. 223, (1989).

[9] R. W. Siegel, Nanostructured Mater. 4, P. 121, (1994).

[10] Chin-Yih. Hong, I. J. Jang, H. E. Horng, C. J. Hsu, Y. D. Yao, and H. C. Yang, J.Appl. Phys. 81, P. 4275, (1997).

[11] M. Grätzel, Nature, 414, P. 338, (2001).

[12] T. Mütschele and R. Kirchheim, Scripta Metall. 21, P. 1101, (1987).

[13] Jianxun Qiux, Le Liang, Mingyuan Gu, "Nanocrystalline structure and magnetic properties of barium ferrite particles prepared via glycine as a fuel", Journal. Materials Science and Engineering, A 393, P. 361–365, (2005).

[14] Jozef Sláma<sup>\*</sup>, "Properties of M-type barium ferrite doped by selected ions", Journal of Electrical Engineering, V. 56, N, 1-2, P. 21-25, (2005).

[15] D Bahadur<sup>\*</sup>, S Rajakumar and Ankit Kumar, "Influence of fuel ratios on auto combustion synthesis of barium ferrite nano particles", Journal. Chem. Sci., V. 118, N, 1, P. 15–21, (2006).

[16] Hsing-I Hsiang \*, Ren-Qian Yao, "Hexagonal ferrite powder synthesis using chemical co-precipitation", Journal of Materials Chemistry and Physics, V, 104, P. 1–4, (2007).

[17] Ping Xu, Xijiang Han,\* and Maoju Wang, "Synthesis and Magnetic Properties of  $BaFe_{12}O_{19}$  Hexaferrite Nanoparticles by a reverse Microemulsion Technique", Journal. Phys. Chem, V. 111, P. 5866-5870, (2007).

[18] R. Nowosielski<sup>a</sup>, "Barium ferrite powders prepared by milling and annealing", Journal, Achievements in Materials and Manufacturing Engineering", V. 22, P. 45-48, (2007).

[19] A.Ataie and S. Jahangir, "Influence of intensive milling on synthesis of barium hexaferrite nano particles", (special Issue of nanotechnology) 9, P. 34-39, (2010).

[20] H.Z. Wang<sup>a</sup>, "Study of formation mechanism of barium hexaferrite by sintering curve ", Journal of Alloys and Compounds,V. 504, P. 70–75, (2010).

[21] P M Prithviraj Swamy, "Barium ferrite nanoparticles prepared by self-propagating low-temperature combustion method and its characterization", Journal. Bull. Mater. Sci., V. 34, N, 7, P. 1319–1323, (2011).

[22] H. Ovalioglu<sup>*a*</sup>, "Magnetic Properties of Nano-Crystalline Barium Ferrite Synthesized by Different Synthesis Route", Journal. ACTA Physica Polonica <sup>a</sup>, N, 5, V, 118, P. 1020-1021, (2010).

[23] M.A Iqbal,amna Mir and Shezad Alam<sup>\*,</sup> "synthesized of nano sized barium hexaferrites ,using sol-gel combustion method", Journal. NSTInanotech, V,1, P. 103-106, (2010).

[24] Q. Mohsen<sup>\*</sup>, "Barium hexaferrite synthesis by oxalate precursor route ", Journal. Alloys and Compounds", V, 500, P. 125–128, (2010).

[25] A.M.Bhavikatti<sup>\*</sup>, "Characterization and electromagnetic studies of nano-sized barium ferrite", International Journal of Engineering Science and Technology, V. 2, 11, P. 6532-6539, (2010).

[26] Jiajie Liu, "Preparation and properties of barium-ferrite-containing glass ceramic fibers via an electrospinning/sol–gel process", Journal. Sol-Gel Sci Technol, V. 61, P. 185–191, (2012).

[27] Widyastuti, "Synthesis of nanoparticle barium hexaferrite by sol gel auto combuion", Journal Teknik Industri, V. 12, N, 2, P. 129-135, (2011).

[28] S. Kanagesana<sup>\*</sup>, "Effect of Microwave Calcinations on Barium Hexaferrite Synthesized via Sol-Gel Combustion", Journal. Sci. Res., Vol. 3 (3), P. 451-456, (2011).

[29] Virk H. S., "Comparative study of Ba-M hexaferrite particles prepared using microemulsion processing and co-precipitation techniques", International Journal of Advanced Engineering Technology, V, II, P. 131-143, (2011).

[30] K. Sadhana, "Structural and magnetic properties of nanocrystalline  $BaFe_{12}O_{19}$  synthesized by microwave-hydrothermal method", Journal. Appl Nanosci, P. 1-6, (2012).

[31] M.J. Molaei, "Synthesis and characterization of  $BaFe_{12}O_{19}/Fe_{3}O_{4}$ and  $BaFe_{12}O_{19}/Fe/Fe_{3}O_{4}$  magnetic nano composites", Journal, Powder Technology, V, 221, P. 292–295, (2012).

[32] Cong-Ju Li<sup>a,b,\*</sup>, "Magnetic and Microwave Absorbing Properties of Electrospun  $Ba_{(1_x)}La_xFe_{12}O_{19}$  Nanofibers", Journal. Magnetism and Magnetic Materials, V, 324, P. 1305–1311, (2012).

[33] M.Wilson, K. Kannangara, G. Smith, M. Simmons, B.Raguse Nanotechnology emerging technologies, Chapman & Hall/CRC, (2002).

[34] J. Cerda, J. Arbiol, R. Diaz, G. Dezanneau and J.R. Morante, "Synthesis of perovskite-type  $BaSnO_3$  particles obtained by a new simple wet chemical route based on a sol–gel process", Journal. Materials Letters, V, 56, P. 131–136, (2002).

[35] J. Behera "Synthesis and characterization of ZnO nanoparticles", MSc thesis, National Institute of Technology, India, (2005).

[36] T. Theivasanthi, M. Alagar "X-Ray Diffraction Studies of Copper Nanopowder", Department of Physics, PACR Polytechnic College, India, (2010). [37] K. Zadeh, B. Fry, "Nanotechnology-Enabled Sensors", springer, Australia, (2008).

[38] C. Raab, M. Simko, U. Fiedeler, M. Nentwich, A. Gazso, "Production of nanoparticles and Nanomaterials", Institute of technology assessment of the austrian academy of sciences, N,6, (2011).

[39] Dti, "Nanoparticle technology: production and application development", A mission to Russia and Ukraine, (2005).

[40] C. Barry; N. Grant "Ceramic materials: science and engineering", Springer,(2007).

[41] K. Flaurance. "Complex oxides of the system Cu-Ni-Fe-O:synthesis

parameters, phase formation and properties", The faculty of mathematics and natural sciences of the dresden university of technology, (2004).

[42] W. Ervens and H. Wilmesmeier, Ullmann's Encyclopedia of Industrial Chemistry, fifth edition, V, A16, P. 1-51, (1990).

[43] C.W. Chen, "Magnetism and Metallurgy of Magnetic Materials", North Holland Publishing Company, (1997).

[44] W.H. Yeadon & A. W. Yeadon, Handbook of small Electric Motors, Mcgraw Hill Company Inc. USA, (2001).

[45] M. V. Rane, D. Bahadur, S. D. Kulkarni & S. K and Date, J. Magn, Magnetic Material 293, P. 1256, (1999).

[46] S. Hirosawa, A. Hanaki, H. Tomizawa and A.Hamamur, Physica B, V, 164, P. 117-123, (1990).

[47] J.D. Livingston, "A review of coercivity mechanisms", Journal. Appl. Phys, P. 522-541, (1981).

[48] Min Chen and David E. Nikles, Nano Lett, P. 211–214, (2002).

[49] F.X.N.M Kools and D. Stoppels, Kirk-Othmer Encyclopedia of Chem. Tech., Fourth Edition10, P. 381-413, (1993).

[50] Carter, C. Barry; Norton, M. Grant, Springer, (2007).

[51] Shriver, D. F., Atkins, P. W., Overton, T. L., Rourke, J. P., Weller, M.T.Armstrong.

[52] E.W. Gorter, Proceeding IEEE, 104B, P. 2255-2257, (1957).

[53] Pooja Chauhan, Master thesis, Thapar University, (2010).

[54] V. Petkov, T. Ohta, Y. Hou, Y. Ren, "Atomic-Scale Structure of nanocrystals by High-Energy X-ray Diffraction and Atomic Pair Distribution Function Analysis Nanoparticles" J. Phys. Chem. C 111, p. 714-720, (2007).

[55] H. Keller, "X-ray Diffraction", University stuttgart fakultat for mathematic and physic, advanced practical course, (2011).

[56] D.Garmo's, "Materials and Processes in Manufacturing", Wiley,10th Edition, (2007).

[57] J.Connolly, " Elementary Crystallography for X-Ray Diffraction", Introduction to X-Ray powder diffraction, Spring, (2012).

[58] U. Ghazanfar, "Preparation and characterization of ferrite materials for practical applications", university of the Punjab,Lahore, Pakistan, (2005).

[59] A. Goldman, "Modern ferrite technology", Van nostrand reinhold, New York, (1990).

[60] Tahseen H. Mubarak, Ph. D thesis, University of Technology, (2003).

[61] James D. Patterson, Bernard C. Bailey, "Solid State Physic Introduction to the Theory", Springer-Verlag Berlin Heidelberg ,(2007), p.(509), (2007).

[62] Jalle, "An outline of polymer chemistry", Oliver and Boyed, Ltd, (1974).

[63] M.C. Lovell, A.J. Aery, and M.W. Vernon, "Physical properties of materials", New York (1976).

[64] R.A. Levy, "Principle of solid state physics", 4th edition, Academic press (1976).

[65] H. Frohlich, "theory of dielectrics", 1st edition, Oxford University Press. (1958).

[66] S. Borowitz, and A. Beiser, "Essentials of Physics", 2nd edition, Addison-Wesley publishing Company, (1971).

[67] William D. Callister, Jr., "Materials science and engineering an introduction". John Wiley & Sons, p.(702-709), (2007).

[68] Brian S. Mitchell, "An Introduction To Materials Engineering And Science", A John Wiley & Sons, Inc, P.(99-100), (2004).

[69] Mailadil t.Sebastian, "dielectric materials for wireless communication", Elsevier, (2008).

[70] Dr .Sobhi S. al-Rawi 'Dr Shaker Shaker 'Dr Maolod Y. Hassan ' "Solid State Physics ", Ministry of Higher Education and Scientific Research, University of Mosul ' p.(329-399), (1988).

[71] B. Tareev, "Physics of dielectric materials", Mir publishers, Moscow, P.(101-145,174-202), (1979).

85

[72] R.S. Khurmi, R.S. Sedha, "material Science", 2nd edition, S. Chand and Company Ltd, (1989).

[73] Zuo-Guang Ye,"Handbook of dielectric, piezoelectric and ferroelectric materials Synthesis, properties and applications ", Wood head Publishing Limited,(2008).

[74] W.D. Gingery, H.K. Bowen, and D.R. Uhlmann, "Introduction to Ceramic", 2nd , Wiley New York, (1976).

[75] D. Halliday, R. Resnik, "Physics", Part (1,2), John Wily and Sons., Inc., (1962).

[76] Anthory, R. West, "Solid state chemistry and application", (1989).

[77] Abdullah A. H. Al-tamimi, "Preparation and Study of The Dielectric Properties of some Epoxy Composite", Master Thesis, College of Education, University of Basrah, (2003).

[78] I.J. McColm, "Ceramic Science for materials Technologists", Leonard Hill, New York, (1983).

[79] D. William, "material science and engineering an introduction" John wiley &son ,Inc , p.676-681, (2000).

[80] S. Feldbauer," Advances in Powder Metal Sintering Technology", Abbott Furnace Company, St. Marys, PA 15857.

[81] A. Verma, T. Goel, R. Mendiratta, R. Gupta" High-resistivity nickel zinc ferrites by the citrate precursor method" Journal of Magnetism and Magnetic Materials 192, p. 271-276, (1999).



Some ASTM cards which are used to match the XRD patterns obtained in this study.

#### Card no. 200127





#### Card no. 280144

🛞 PDF # 280144, Wavelen	gth = 1.54056 (A)	<u>- 🗆 ×</u>
28-0144 Quality:	Ba5 Fe14 026 Barium Iron Oxide	-
Molecular Weight: 1884.49	Ref: Appendino, P., Montorsi, Ann. Chim. (Rome), 63, 449 (1973)	
Volume[CD]: Dx: Dm:	0	
S.G.:	ta a ta	
, Cell Parameters: - a b c	3.20 3.20	
α β γ		
SS/FUM: F = [ , ]   I/Icor:		
' Rad: CoKa Lambda: 1 7902	0 10 20 30 40 50 60 26°	
Filter: Fe	28 int-v h k l 28 int-v h k l 28 int-v h	k I
a-sp:	5.417 2 28.680 100 41.304 10 16.341 6 29.868 62 42.738 15	
	17.070 3 30.063 37 43.938 30 17.905 5 20.762 6 44.902 29	
	18.947 13 31.305 7 45.425 8	
	19.846 6 32.363 90 46.084 24 20.258 14 32.839 7 46.890 10	
	20.542 6 33.279 5 49.325 8 20.884 4 34.562 72 51.563 11	
	21.446 5 34.798 30 53.044 14	
	21.819 15 35.495 10 54.546 22 23.205 4 36.070 4 54.864 28	
	24.032 5 36.480 4 55.078 22 25.354 5 36.993 9 56.364 23	
	25.802 18 37.702 16 57.754 29	
	26.033 55 38.404 20 58.274 12 26.506 14 38.887 11 59.011 16	
	26.749 9 39.672 16 62.119 25 27.080 38 40.301 11 62.773 17	
	27.769 58 40.912 28 63.202 10	
		-



#### Card no. 261136

😨 PDF # 261136, Wavelen	gth = 1.5	4056 (/	4)										×
26-1136 Quality: CAS Number: 1317-61-9	Fe3 04 Iron Oxi	de lo et al	L Gev	ophus	Peo 79	1165 (1	974)						-
Molecular Weight: 231.54 Volume[CD]: 529.53 Dx: 5.809 Dm: 5.150 S.G.: Fd3m (227) Cell Parameters:	sity ->			<u>, projo</u> ,		,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,	<u></u>			10			
a 8.090 b c α β γ SS/FOM: F 9=84(.0082, 13)	Variable Inten			1		. T							
Rad:	0		15		30	45		60	7	5 20°			
Lambda: Filter:	20	int-v	h l	< 1	20	int-v	h l	< 1	20	int-v	h	k I	
o-sp:	18.988 31.249 36.820	5 26 100	1 2 2 3	1 2 0   1	38.524 44.762 55.622	10 36 30	2 : 4 : 4 :	220022	59.303 65.185 77.249	63 85 10	5 4 5	1 1 4 0 3 3	
													-

🛞 PDF # 200126, Wavelen	igth = 1.54056 (	(A)		
20-0126 (Deleted)	Ba Fe 02.50	de		•
LAS Number: Molecular Weight: 233.18	Ref:			
Volume[CD]: 67.77 Dx: 5.713 Dm:	•	1		
S.G.: Cell Parameters:	sity slit			
a 4.049 b 4.049 c 4.139	able			8
α 89.68 β 89.68 γ 92.8 SS/FOM: F23=32(0.026, 28)			The Dirac Jack State	8
Micor:		15 30	45 60	26°
Lambda: 1.54056	, in the second s		40 00	2.0
Filter:	20 int-v	h k   28	int-v h k l 20	int-v h k l
a-sp.	8.334 2	30.949	100 1 0 1 54.196	$ \begin{array}{cccccccccccccccccccccccccccccccccccc$
	21.983 18	0 1 0 36.449	7 55.006	
	25.802 3	37.554	12 1 1 1 56.214	14 2 1 1
	26.914 7	38.992	13 1 1 56.515	
	29.062 4	44.762	36 2 0 0 64.177	8 0 2 2
	29.355 6	1 1 0 49.296	6   64.576 9 1 0 2   66.978	4 2 2 0
	30.796 95	0 1 1 49.611	6 0 1 2	
				-

#### Card no. 011053

PDF # 011053, Wavelen	gth = 1.54056 (A)			- 🗆 ×
01-1053 (Deleted) CAS Number: Molecular Weight: 159.69	Fe2 03 Iron Oxide Ref: Hanawalt, et al., Anal.	Chem., 10, 475 (1938)		<u> </u>
Volume[CD]: 300.60 <u>Dx: 1.764 Dm: 5.260</u> S.G.: R3c (167) Cell Parameters:	stry +	111		
a 5.028 b c 13.73 α β γ SS/FOM: F23=7(0.055, 60)	Variable	ไปปี มีเลี้ยว เ	132.9	
Rad: MoKa Lambda: 0.709	0 25	50 75 100	125 20°	
Filter: ZrO2 d-sp: Minoral Namo:	28 int-v h k l 24.164 13 0 1 2	29 int-v h k l 64.177 93 3 0 0	20 int-v h k 88.895 24 2 2	6
Hematite	35,743         80         1         1         0           40,990         22         1         1         3           49,496         92         0         2         4           54,231         100         1         1         6           57,557         22         1         2         2	72.030         37         1         0.10           75.372         28         2         1         7           77.547         7         3         0         6           80.676         18         3         1         2           83.217         12         0         210         1	33.217         20         2         1           106.71         28         3         2           108.35         14         4         1           117.71         9         3         0           122.16         15         4         1           132.98         5         1         0	4 0 2 6 6
	62.258 90 2 1 4	85.015 31 1 3 4		
				-



😵 PDF # 021047, Wavelen	gth = 1.54	4056 (A	4)												_	
02-1047 (Deleted)	y-Fe2 03	3														-
LAS Number: Molecular Weight: 159.69	Ref: Brit	tish Muse	eum	(Na	atura	al History)					F	2				
Volume[CD]: 579.05											2	6				
Dx: Dm: 4.590	=															
Cell Parameters:	e SI									-						
a 8.335 b c	Inte															
SS/FOM: F 9=2(0.149, 26)	S							ΞŤ.	2							
I/Icor: Bad: MoKa1	0	1	0		20	30	4	0	5	0	60	20°				
Lambda: 0.709	20					20				1.00			E.			
Filter: d-sp:	20	int-v	n	ĸ	.	41,000	Int-V	n	к I о 1	20 E4 00	-	Int-v	n A	ĸ	2	
	30.484	20	3	1	ŏ	43.692	50	4	δo	57.55	57	74	5	1	í	
	35.743	53	3	1	1	49.496	16	4	2 1	62.72	26	100	4	4	0	
					~											-
## الخلاصية

يتضمن هذا البحث تحضير باريوم فرايت (BaFe<sub>12</sub>O<sub>19</sub>) بأحجام نانوية مختلفة كمادة مغناطيسية ودراسة بعض الخواص الكهربائية كثابت العزل الكهربائي، ظل الفقد، الفحص بالأشعة السينية للمادة المنتجة. تم تحضير مسحوق الباريوم فرايت النانوي باستخدام طريقة الترسيب الكيميائي (co-precipitation) باستخدام المحلول المائي ومحلول (ايثانول \ ماء) لكلوريد الحديد الكيميائي (FeCl<sub>3.</sub>6H<sub>2</sub>O) باستخدام المحلول المائي ومحلول (ايثانول \ ماء) لكلوريد الحديد ويجفف الباودر المترسب في كلا الحالتين عند درجة 80 م° لمدة 12 ساعة، بعدها يلبد في درجات ويجفف الباودر المترسب في كلا الحالتين عند درجة 80 م° لمدة 12 ساعة، بعدها يلبد في درجات محرارة مختلفة لمدة ساعتين. ولوحظ من خلال فحص SEM ان جزيئات فرايت الباريوم النانوية ويجفف البود نتائج الفحوصات على العموم تناقص في ثابت العزل الكهربائي مع زيادة التردد ، وأن قيم ثابت العزل الكهربائي للنماذج المحضرة باستخدام الماء هي أعلى من قيم ثابت العزل النماذج المحضرة باستخدام محلول (ايثانول\ ماء) بفارق قليل جدا وهذا يعزى إلى امترك الماء شوائب عديدة ويحتاج الى حرارة معينة ليتم التخلص منه مما ينتج عنه ثابت عزل الماء شوائب عديدة ويحتاج الى حرارة معينة ليتم التخلص ما ماء هي أيات العزل موائب عديدة ويحتاج الى حرارة معينة ليتم النخلص ماهما ينتج عنه ثابت عزل كهربائي عالي أما الإيثانول فهو يمتاز بأنه محلول متطاير لذا فانه لا يؤثر على عزلية المادة.

بينت النتائج لكلا النموذجين تناقصاً في ثابت العزل الكهربائي مع زيادة درجة الحرارة وزيادة التردد. أما ظل الفقد فلوحظ بأن قيمته تقل مع زيادة الترددات. أما فحوصات الأشعة السينية لكلا النموذجين فقد وضحت ان هناك تغير بالأطوار عند زيادة درجة الحرارة لكل نموذج.

أما صور المجهر الالكتروني الماسح فقد بينت ان الحبيبات ذات اشكال واخرى غير منتظمة، وهي نتيجة مماثلة لما توصل اليه باحثون آخرون. وقد تم استخدام قانون الكثافة لحساب الكثافة الحجمية وتبين إن الكثافة وانكماش العينات يزداد بازدياد درجة الحرارة. ومن المعروف ان مقاومة الفرايتات تعتمد على نقاوة المواد الاولية وعلى تفاصيل عملية التحضير كدرجة الحرارة وانه من المتوقع يزداد بازدياد درجة حرارة الكلسنة.

جمهورية العراق وزارة التعليم العالي والبحث العلمي جامعة ديالى كلية العلوم/قسم الفيزياء



## تحضير فيرايت الباريوم النانوي ودراسة خصائصه الفيزيائية

رسالة مقصة إلى مجلس كلية الملهم – جامعة حيال هي في جن من متطلبات نيل حرجة الماجستين ملهم في الفيزيل تحمما نسرين زيدان خلف بإهران أ.م.د. تحسين حسين مبارك أ.د.كريم هنيكش حسن 1435هـ