International Journal of Mathematics and Physical Sciences Research ISSN 2348-5736 (Online)
Vol. 7, Issue 2, pp: (105-114), Month: October 2019 - March 2020, Available at: www.researchpublish.com

SYNTHESIS OF NI-CO NANO FERRITE USING SOL-GEL METHOD

HITESH PATIL

Department of Physics

Modern College of Arts, Commerce and Science, Ganeshkhind, Pune, Maharashtra, India.

Author email id:hitesh.patil4u@gmail.com

Abstract: **In modern world, the technology is rising upwards at large scale and the leading the day is Nano-Technology. Hence it appealed to us that we should contribute to the science with our little efforts.**

Nickel Nano-ferrites i.e. NiCo Nanoparticles were prepared by using sol-gel synthesis method using Ni(NO3)2 .6H20 (Nickel Nitrate), Fe (NO3)3 .9H20 (Ferric Nitrate),Cobalt Nitrate Co(No3)2.6H20 (C6H807.H20 (Citric Acid) as fuel agent, NH3 (Ammonia) as pH balancing agent and followed by calcinations at certain temperature. The pH of three samples prepared were varied in order to understand changes in properties. Multiple techniques (XRD, 2 Probe) were commanded to characterize the crystal structures, electrical properties and magnetic properties of NiCo.

Keywords: **Nano-Technology, Nano-ferrites, NiCo, pH, characterize.**

1. INTRODUCTION

1.1 Introduction to Nanotechnology:

Nanotechnology is defined as the "Engineering of functional systems at the molecular scale". Simply defined, "Nanotechnology is the creation, use or manipulation of matter on the atomic scale". Nanotechnology is an emerging, interdisciplinary field combining principles of chemistry and physics with the engineering principles of mechanical design, structural analysis, computer science, electrical engineering, and system engineering. Built to atomic specification, the products would exhibit order of magnitude improvements in strength, toughness, speed, and efficiency, and be of high quality and low cost. Nanotechnology is the technology of preference to make things small, light and cheap.

Nanotechnology is the study and design of systems at the nanometer scale [0.000000001(10^9) meter] the scale of atoms and molecules. Human ability to manipulate materials on the nanoscale could revolutionize the way that almost everything is designed and made as nature does it. The field of Nanometer Scale Science and Technology (NSST) is very broad, ranging from nanoparticles, nanoclusters, and mesoscopic systems to individual atoms and molecules and their self-assembly into defined structures such as nanowires or biomolecules. This is a field where the border between the traditional disciplines like physics, chemistry and biology is no longer detectable and interesting synergies arise, e.g., instruments developed in physics provide the precision and sensitivity to perform specific molecular recognition experiments in biology.

Nanometer-scale traps will be constructed that will be able to remove pollutants from the environment and deactivate chemical warfare agents. Computers with the capabilities of current workstations will be the size of a grain of sand and will be able to operate for decades with the equivalent of a single wristwatch battery. Robotic spacecraft that weigh only a few pounds will be sent out to explore the solar system, and perhaps even the nearest stars. Nanotechnology will change the nature of almost every human made object. The total societal impact of nanotechnology is expected to be greater than the combined influences that the silicon integrated circuit, medical imaging, computer-aided engineering, and manmade polymers have had in this century. Significant improvement in performance and changes of manufacturing paradigms will lead to several industrial revolutions in the twenty first century.

Nanometer length scale: Nanometer (nm) is one-billionth of a meter. For comparison, a single human hair is about 80,000 nm wide, a red blood cell is approximately 7,000 nm wide and a water molecule is almost 0.3nm across. Why this length scale is so important? There are five reasons why this length scale is so important:

I. The wavelike properties of electrons inside matter are influenced by variations on the nanometer scale. By patterning matter on the nanometer length scale, it is possible to vary fundamental properties of materials (for instance, melting temperature, magnetization, charge capacity) without changing the chemical composition.

II. The systematic organization of matter on the nanometer length scale is a key feature of biological systems. Nanotechnology promises to allow us to place artificial components and assemblies inside cells, and to make new materials using the self-assembly methods of nature. This is a powerful new combination of materials science and biotechnology.

III. Nanoscale components have very high surface areas, making them ideal for use in composite materials, reacting systems, drug delivery, and energy storage. 0

IV. The finite size of material entities, as compared to the molecular scale, determine an increase of the relative importance of surface tension and local electromagnetic effects, making nanostructure materials harder and less brittle.

V. Comparable to the material entity size, making materials suitable for various optoelectronic applications.

The nanoparticles that we have synthesized are Nickel Nano Ferrites (NiCo) since we have used ferromagnetic materials as our reactants. It is clear that the product has electrical properties.

The basic idea behind our project is to check the variation in the properties of nanomaterials such as electrical properties , crystal structure etc. with variation is the pH as we synthesize them by sol-gel method. And with the help of that data determining the uses and further applications in respective fields. We have successfully synthesized 3 batches of nickel-nano ferrites (NiCo) as with varying pH .

Ferrites:

Ferrites are electrically non-conductive ferrimagnetic ceramic compound materials, consisting of various mixtures of iron oxides such as Hematite (Fe203) or Magnetite (Fe304) and the oxides of other metals. Ferrites are most important ferrimagnetic substances, which contain certain double oxides of iron and another metal. The general form of ferrite is MFe204, where M represents a divalent metal ion; generally, transition metal ions like Mn, Fe, Co, Ni, Cu, and Zn. Ferrites are predominantly ionic and have very stable crystal structure. Great majority of ferrites contain iron oxides as major constituents but there are some ferrites based on Cr, Mn and other elements. Ferrite cores are used in electronic inductors, transformers, and electromagnets where the high electrical resistance of the ferrite leads to very low eddy current losses. They are commonly seen as a lump in a computer cable, called a ferrite bead, which helps to prevent high frequency electrical noise (radio frequency interference) from exiting or entering the equipment.

2. SYNTHESIS TECHNIQUES

2.1 Physical Methods:

There is a large number of techniques available to synthesize different types of nanomaterials in the form of colloids, clusters, powders, tubes, rods, wires, thin films, etc. Some of the already existing conventional techniques to synthesize different types of materials are optimized to get novel nanomaterials and some new techniques are developed. In this chapter, we will be looking at some Mechanical Methods to obtain nanomaterials.

I. Mechanical Methods

II. Methods Based On Evaporation

III. Sputter Deposition

IV. Chemical Vapor Deposition (CVD)

V. Electric Arc Deposition

VI. Ion Bean Techniques (Ion Implantation)

2.2 Chemical Methods:

There are numerous advantages of using chemical methods to synthesize nanomaterials. In some cases, nanomaterials are obtained in the form of colloidal particles in solutions, which can be filtered and dried to obtain powder. In some methods we can obtain thin films or nonporous materials by electrodeposition, etching etc. Advantages of Chemical Methods are manifold.

I. Colloids and Colloids in Solutions

II. Growth of Nanoparticles

III. Synthesis of Metal Nanoparticles by Colloidal Route

IV. Synthesis of Semiconductor Nanoparticles by Colloidal Route

V. Langmuir-Blodgett (LB) Method

VI. Micro emulsions

VII. Sol Gel Method

VIII. Hydrothermal Synthesis

IX. Sonochemical Synthesis

X. Microwave Synthesis

2.3: Biological Methods

Many of the materials synthesized by microorganisms, animals and plants in nature can indeed be synthesized using them in laboratories been on large scale. This is considered very attractive possibility so as to have eco-friendly or so-called green synthesis.

I. Synthesis Using Microorganisms

II. Synthesis Using Plant Extracts

III. Use of Proteins, Templates like DNA, S-Layers etc.

Iv. Synthesis of Nanoparticles Using DNA

3. SYNTHESIS OF NICKEL NANO FERRITES (NICO)

3.1 Methods of synthesis of Ferrites:

Preparation of nanomaterials can be divided into two broad spectrum top down and bottom up, each of which has two directions physical and wet chemical. The most important criteria for preparation of nanoparticles are: Proper size with narrow size distribution well dispersed particles, equiaxial shape of particles, high purity, and homogeneous composition. Most of the wet chemical methods have common feature that the mixing of components takes place at the atomic or molecular scale. Some of the non-conventional processes are: Sol-gel method, Co-precipitation method, Precursor method, combustion method, Hydrothermal, Spray drying. Etc.

3.2 Sol-gel Method:

In general, Sol-Gel process consists of following steps:

I. Preparation of homogeneous solution of either by dissolution of metal organic precursors in an organic solvent that is miscible with water, or by dissolution of inorganic salts in water.

II. Conversion of the homogeneous solution into a sol by treatment with a suitable reagent (Generally water with or without any acid/base).

III. Maintaining the pH as solution by using a liquid (ammonia liquid).

IV. By using continuous heat treatment, the formation of gel.

3.3 Raw materials/chemicals -

Raw materials/chemicals used for the synthesis of nickel nano ferrites are as follows:

Table 1: List of chemicals/materials required.

Name	Chemical Formula	Molecular Weight
NICKEL NITRATE	Ni(NO3)2.61120	290.81
FERRIC NITRATE	Fe(NO3)3.91120	404.00
COBALT NITRATE	Co(No3)2.6H20	291.03
CITRIC ACID	C6H807 .H20	192.13

3.4 Material Expansion-

NICKEL NITRATE:

Nickel nitrate Ni(NO3)2 the anhydrous form is not commonly encountered, thus "nickel nitrate" usually refers to nickel (II) nitrate hexahydrate. The formula for this special is Ni (NO3)2 .6H20 The formula indicates that the nickel center is surrounded by six water molecules in this hydrate salt. The nitrate anions are not bonded to nickel. The 6H20 represents water of crystallization.

Table 2: Physical properties of nickel nitrate.

Molar Formula	Ni(NO3)2
Molar Weight	290.81 g/mol
Melting Point	56.7oC
Odor	Odorless
Density	2.05 g/cm3(hexahydrate)
Boiling Point	136.7oC
Appearance	Emerald green hygroscopic solid
Solubility	Soluble in ethanol

FERRIC NITRATE:

Iron (III) Nitrate or Ferric Nitrate is a chemical compound with molecular formula Fe (NO3)3 since it is deliquescent. It is commonly found in its non-hydrate form Fe (NO3)3 .9H20.in which it forms colorless to pale violet crystals.

Table 3: Physical properties of ferric nitrate.

Molar Formula	-	**Fe(NO3)3**
Molar Weight	-	**440.00**
Melting Point	-	**47.20 C**
Appearance	-	**Pale Violet Crystal**
Solubility in Water	-	**Very soluble**

Cobalt Nitrate:

Cobalt Nitrate is the inorganic cobalt (II) salt of nitric acid, often with various amounts of water. It is more commonly found as a hexahydrate , Co(NO3)2.6H20, which is a red-brown deliquescent salt that is soluble in water and other polar solvents.

Molar formula	**Co(No3)2**
Molar weight	**291.03**
Melting point	**55 °C**
Appearance	**Red crystalline**
Solubility in water	**Soluble**

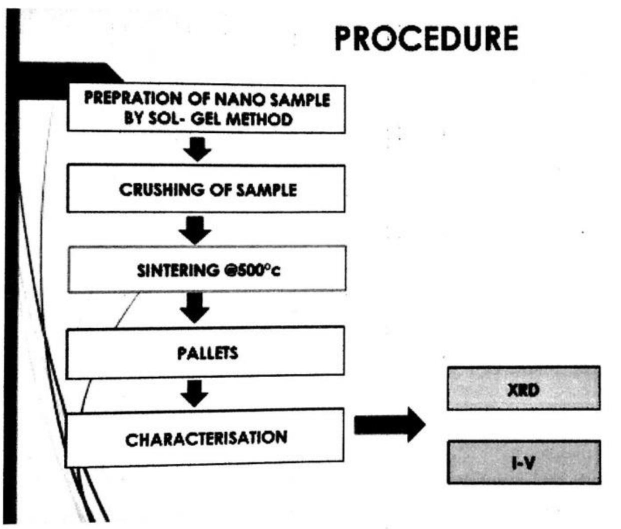
CITRIC ACID:

It is weak organic acid. It is a natural preservative and is also used to add an acidic or sour, taste to foods. In biochemistry, the conjugate base of citric acid, citrate is important as an intermediate in the citric acid cycle and therefore occurs in the metabolism of virtually all living things.

Table 4: Physical properties of citric acid.

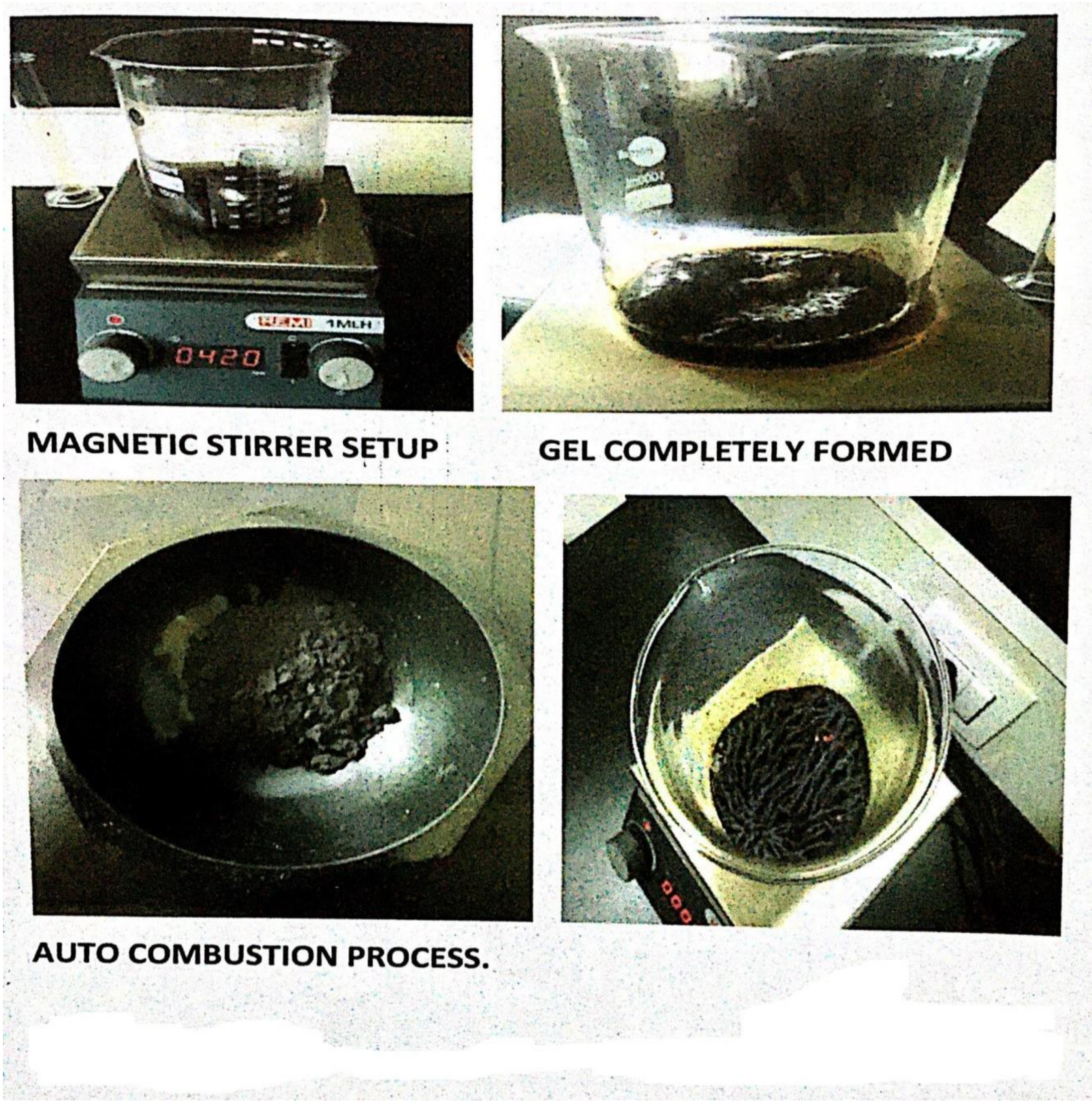
Molecular Formula	**C6H807**
Molecular Weight	**192.12**
Melting Point	**1530C**
Appearance	**White Crystalline Solid**
Solubility in Water	**73gm/100m1**

EXPERIMENTAL PROCEDURE:



CALCULATIONS:

X	Ni (gm)	Co (gm)	Fe (gm)	Citric acid(gm)
0.1	5.58	0.62	17.23	12.29



After some time as the temperature of the beaker decreases the residue as seen in above image is then later grinded finely. The powder is then used for various characterizations. By mixing binder solution with the powder and applying external pressure of 3 tons with the help of hydraulic press a pallet is made, which is later used for further analysis and characterization.

4. CHARACTERISATIONS AND ANALYSIS

4.1 XRD Characterization:

1.1 Structural characterization using powder X-ray diffraction:

X-ray diffraction technique is used to study the following aspects.

I. For phase analysis.

II. Determination of crystal size.

III. Determination of crystal lattice.

IV. Study of crystal distortion by stress.

1.2 Principle:

X-rays are electromagnetic radiation of wavelength ~ 1Ao (10-10m). X-rays are produced when high energy charged particles collide with matter. In this process, electrons are ejected from the core shell around the nucleus and another electron of higher energy from the outer shells fills the resulting hole. These electrons give up their excess energies in the form of x-rays. If the electron jumps from the L-shell, then it gives Ka radiation or if it jumps from the next higher shell it will gives to Kβ radiation. X-rays for diffraction are usually generated in an evacuated & selected tube by applying a high voltage (30-60 kV) between a cathode (usually tungsten) and a selected anode such as copper. X-rays leaves the tube through windows made of beryllium. This must be adequate cooling of the tube to remove the heat generated. For analytical purposes monochromatic beam of x-rays are used.

1.3 Bragg's Law:

Bragg pointed out that scattering of x-rays by crystals could be considered as reflection from successive planes of atoms in the crystal as shown in figure. Reflection of x-rays can take place only at a certain angle, which are determined by wavelength of x-rays and the distance between the planes in the crystal, and the angle of diffraction is known as Bragg's equation which is given by

$$n\lambda = 2d \sin(\theta)$$

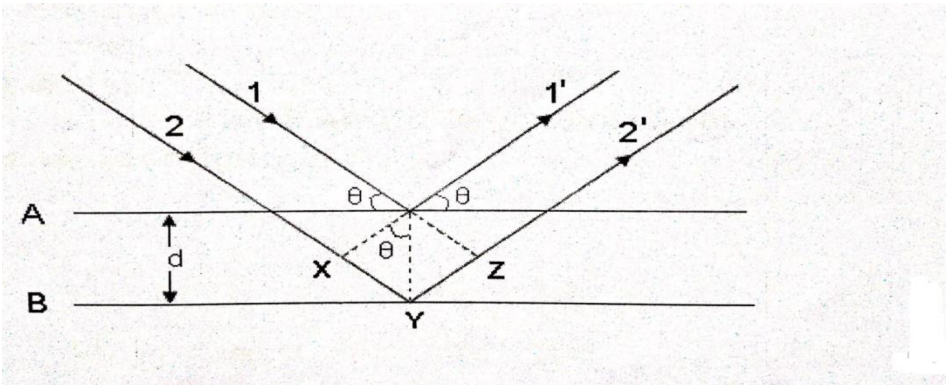


Fig : A and B are two adjacent planes of the crystal, d is the distance between the planes, 1 and 2 are incident rays in the angle of incidence, 1' and 2' are reflected rays. X-ray diffraction pattern is a set of lines or peaks, each of different intensity and position (usually measured as 20). It is known that the width of the diffraction peak increasing when the crystallite size is reduced below a certain limit (< 100nm). Therefore, XRD pattern can be used to estimate the average size of very small crystallites, from the measured width of the peaks in the different pattern. The commonly accepted formula for the determination of crystallite size from XRD line broadening is the Scherer's

1.4 Formula:

$$t = \frac{0.9\lambda}{\beta\cos\theta}$$

Where,

λ =Wave length of X-ray

t =Particle size.

θ= Bragg angle

β= FWHM of the peak and corrected or instrumental broadening.

1.5 XRD pattern of Nickel Ferrite nanoparticles:

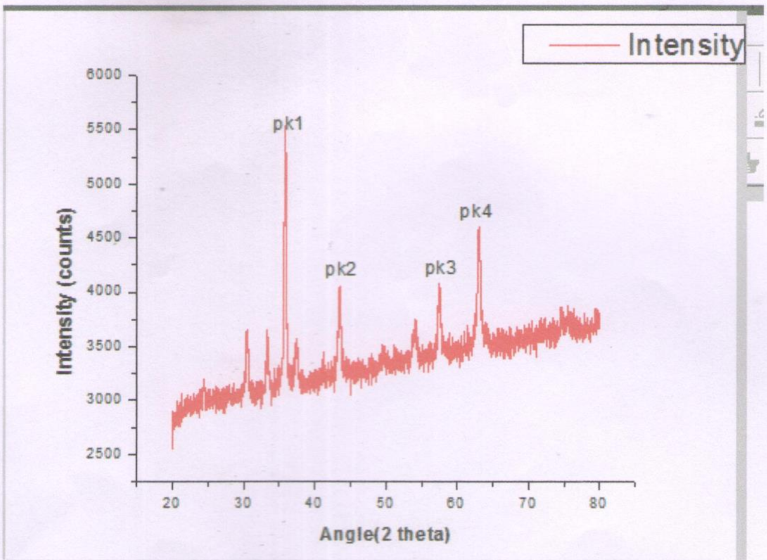


Fig. : XRD of N1

X=0.1,pH 5	θ	Cos(θ)	β	β in radian (10-3)	t=0.91×λ/βcos(θ) (nm)
N1	31.552	0.90	0.22	3.8	31.10

I-V Characteristics and Resistivity:

I-V Characteristics and the resistivity data is used for following aspects:

I. Determination of current and voltage relations about the desired material at variety of temperatures.

II. Determination of the resistance of material over variety temperatures.

III. Determination of conductivity of material.

1.2 Principal:

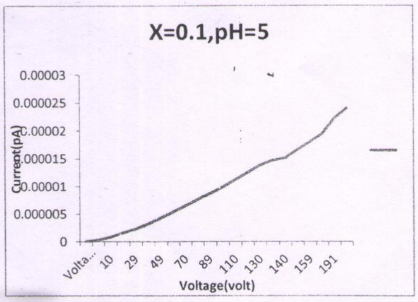
Materials are often classified according to their ability to let current flow through them. Conductivity is defined in terms of properties of electrons (number, effective mass, scattering etc.) in the solids and is given by $\sigma = N e 2\tau/m$ where σ= electrical conductivity, N= no. of electrons, e electron charge, τ =relaxation time, m= mass of electron. Resistivity is the inverse of conductivity. Metals are characterized by very low resistivity. Semiconductors have medium resistivity and insulators have large resistivity. The resistivity in solids can be measured in principle by connecting electrically conducting wires to solid material of known geometry, applying a voltage difference across it and measuring the current flowing through it.

A **current-voltage characteristic** or **I-V curve** (current voltage curve) is a relationship, typically represented as a chart or graph, between the electric current through a circuit, device, or material, and the corresponding voltage, or potential difference across.

Sample > x= 0.1 , PH =5
Thickness = 0.00322 meter
Diarneter =
Crossectional aera = 0.000000785

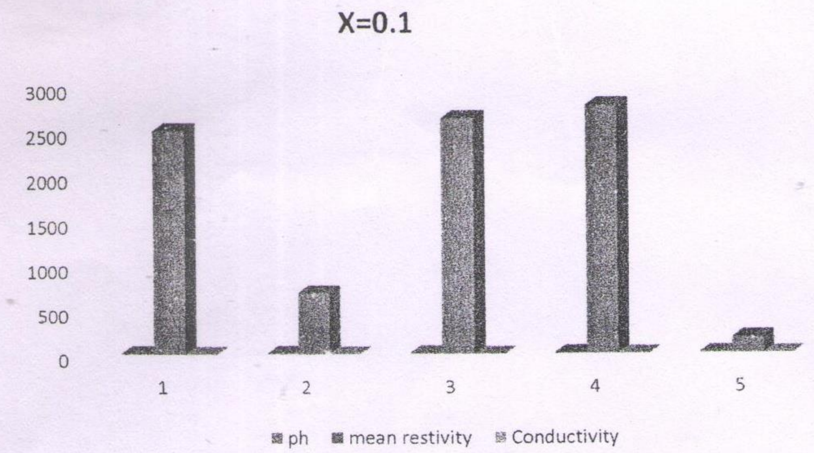
S.No.	Temperature(°C)	Voltage (V)	Current (pA)	Resistance(Ω)	Cross sectional Area(A)	Thickness(L)	Restivity(ρ)	Mean Restivity
1	201	0	3E-07	0	0.000000785	0.00322	0	
2	203	10	8E-07	12500000	0.000000785	0.00322	3047.360248	
3	203	20	1.6E-06	12500000	0.000000785	0.00322	3047.360248	
4	204	29	2.3E-06	12608696	0.000000785	0.00322	3073.859118	
5	204	41	3.2E-06	12812500	0.000000785	0.00322	3123.544255	
6	205	49	4.3E-06	11395349	0.000000785	0.00322	2778.058685	
7	204	58	5.4E-06	10740740	0.000000785	0.00322	2618.472329	
8	205	70	6.5E-06	10769231	0.000000785	0.00322	2625.418116	
9	205	80	7.6E-06	10526316	0.000000785	0.00322	2566.198155	
10	206	89	8.7E-06	10229885	0.000000785	0.00322	2493.931592	
11	205	100	9.9E-06	10101010	0.000000785	0.00322	2462.513307	2501.102603
12	205	110	1.12E-05	9821429	0.000000785	0.00322	2394.354585	
13	206	120	1.25E-05	9600000	0.000000785	0.00322	2340.372671	
14	206	130	1.38E-05	9420290	0.000000785	0.00322	2296.561382	
15	206	137	1.47E-05	9319728	0.000000785	0.00322	2272.045491	
16	206	140	1.51E-05	9271523	0.000000785	0.00322	2260.293651	
17	206	150	1.66E-05	9036144	0.000000785	0.00322	2202.910882	
18	208	159	1.8E-05	8833333	0.000000785	0.00322	2153.467828	
19	206	170	1.94E-05	8762886	0.000000785	0.00322	2136.293637	
20	206	191	2.22E-05	8603604	0.000000785	0.00322	2097.462466	
21	205	200	2.4E-05	8333333	0.000000785	0.00322	2031.573418	

resistivityρ=2501.102603(Ωm)
Conductivity(σ)=1/ρ=0.000399(Ω^-1m^-1)



X=0.1

sr.no	ph	mean restivity	Conductivity
1	5	2501.102603	0.000399
2	6	688.3677376	0.00145
3	7	2639.475318	0.0003788
4	8	2778.836114	0.000359
5	9	179.0591217	0.00584



5. CONCLUSION

Ni-Co Nano-sized powders were synthesized by Sol-Gel combustion method. Sol-gel auto combustion is a unique combination of the combustion and the chemical gelation processes.

1.The Analysis of XRD state that the X=0.1 increase in pH level of the material results in increase in the size of particle.

2. With X=0.1 increase in pH value the conductivity decreases.

Application:

1. Having porous nature and magnetic properties it can used in magnetic sensors.

2. They can be used magnetic storage devices.

3. Coatings, nano -wires , nano- fibers and textiles.

REFERENCES

[1] Corriu, R.J.P, Leclercq. D.: Recent Development of Molecular Sol-gel process. Angew. Chem. Int. Ed. 35, 1420-1436. 1996.

[2] Nanotechnology Principles and Practices by S. K. Kulkarni.

[3] Molecular Structure & spectroscopy by G. Aruldhas.

[4] Fundamental of Molecular Spectroscopy by C. N. Banwell.

[5] Solid State Physics by Reaf & Melford.

[6] Nano Materials by A.K. Bandhyopadhyay

[7] www.researchgate.com

[8] www.academia.edu

[9] Introduction to X-Ray Powder Diffraction Data Analysis by Scott Speakman, Ph.D. Center for Materials Science and Engineering at MIT.

[10] Guidelines for UV-vis Analysis by Nano Composix.

[11] www.wekipedia.org