Facile synthesis of Ni-Cs-Zn ferrite by solution combustion method and characterization

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For the partial fulfilment of the award of degree of

Master of Science in Chemistry (Honours)

By

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DECLARATION

I hereby declare that the dissertation report entitled, "Facile synthesis of Ni-Cs-Zn ferrite by solution combustion method and characterization" submitted for partial fulfillment of the requirement of award of degree of Master of Science in Chemistry (Honours) is entirely my original work and all the ideas and references have been duly acknowledged. It does not contain any work for the award of any other degree or diploma, other than described, at any other university.

Atulika Sharma

Registration number: 11507183

CERTIFICATE

This is to certify that the dissertation report, 'Facile synthesis of Ni-Cs-Zn ferrite by solution combustion method and characterization' submitted by Atulika Sharma for the partial fulfillment of the requirement of award of the degree of M.Sc. honours Chemistry from School of Chemical Engineering and Physical Sciences, Lovely Professional University, Phagwara. is a record of candidate's own work carried out by her under my supervision and guidance. To the best of my knowledge the present work is the result of her original investigation and study.

Signature of supervisor

Dr. Harmanjit Singh Dosanjh

Designation: Assistant Professor

Lovely Professional University,

Phagwara, Punjab, India

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Atulika Sharma

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INTRODUCTION

Ferrites are the magnetic materials or magnetic oxide having composition of Fe, Mg, Ni, Zn. Name ferrite is derived from Latin word "ferrum" i.e. called iron. Ferrites are the good materials then metals so that they are used widely due to their numerous applications in practical field. Ferrites are classified as spinal and inverse spinal, Also the presence of magnetic ferrites are observed naturally (Fe_3O_4) i.e. iron oxide was the first naturally occurring magnetic material that was invented by human, presence of naturally occurring ferrites are in the form of ores and the ore of Fe_3O_4 is called magnesia as a common name and scientifically it called magnetite.

The electrical and magnetic properties of ferrites are most probable area of research in the field of ferrites, because it enhances the development in science and technology [1]. In the microwave material technology, memory cores of computers, radar, satellite communication, ferro fluids, ferro salts, digital recording, nano chips, permanent magnets and a very good chemical stability, low electrical losses ability [2], These are the fields where the demand of ferrites are quite high, In the field of electrical appliances researchers substitute the synthesized ferrites with transition metals oxides [3]. During these days main focus on the development of innovative routes for preparation of ferrites as like MFe_2O_4 at nano scale is in process (Here M may be Mn, Zn,Ni, Mg etc) . According to the latest data demand of soft ferrites are increased by these days i.e. reported by China [4]. Magnetic nano particles are considered as the most probable research area due to their wide range of applications in the field of electronics, high density magnetic recording applications, core materials for power transformers in electronics and tele communication, antenna rods, e.g. Cobalt ferrites ($CoFe_2O_4$) are the widely considered for their property of retaining the chemical stability and good mechanical hardness [5]. The success of its particular applications is depending on its composition and crystal size with a single domain limit [6]. Crystal size is intensively effected by the chemical composition, solution chemistry and precipitation methods [7]. The Condition to get a perfect crystal size is a crystal must have coercivity and magnetization at nano scale. Cobalt ferrites are not essential for its magnetic properties only but also for the catalytic properties which depend on the textual and morphological characterization [8]. Such ferrites are come under the category of spinals but they show the high value of coercivity inversely from the rest of ferrites. Cobalt ferrites are called hard ferrites due to their high coercivity. Cobalt ferrites neither have spinal not inverse spinal structure they have partially inverse spinal structure

[9]. They have coercivity value 1000Oe and moderate magnetization value of 50 emu/g. Due to their such wide range of properties they are the extensively used as ferro fluids in magnetic diagnosis, magneto-mechanical and torque sensors, ferrites are very hard, brittle, chemically inert [10].

TYPES OF FERRITE (MAGNETIC MATERIALS)

Ferrites are classified in two categories on the basis of their magnetic materials.

1) Soft ferrites

2) Hard ferrites

SOFT FERRITES:

Nano Ferrites with low coercivity value are called soft ferrites, Low coercivity value explains that they can be easily reverse in direction without any loss in energy in b-h curve, Since they have low coercivity value so they can be easily magnetized and demagnetized. Soft ferrites are preferred over other electromagnetic materials because they have high value of electrical resistivity which leads to low eddy currents even at high frequency range. The most common soft ferrites are manganese-zinc (Mn-Zn), Cobalt-Zinc(Co-Zn), Nickel-zinc (Ni-Zn), $Co_{(x)}Zn_{(1-x)}Fe_2O_4$ and $Ni_{(x)}Zn_{(1-x)}Fe_2O_4$ respectively. They Possess high resistivity value and higher permeability value. Soft Ferrites are used in transformer or electromagnetic cores made up of nickel, zinc, and manganese compounds. Few of the low frequency applications of soft ferrites are magnetic recording head, inductor, and transformer and filter cores.

The characterization of soft ferrites is done on following basis:

1) Soft ferrites are leftover with the coercive value even after the value of applied external field is removed on the material.

2) In soft ferrites saturation flux can be induced.

HARD FERRITES:

Nano ferrites having magnetic hardness are called hard ferrites. This hardness is mainly due to the fine particles having shape and crystalline anisotropy. A large value of anisotropy is the characteristics of hard ferrites, hence large value of coercivity are considered as the inherent property of hard ferrites. Coercivity Value of hard ferrites are 3000 Oe times as compare to the other ferrites. Hard ferrites are ferromagnetic nature and are used to make the permanent magnet. Barium and Strontium are widely studied as hard ferrites [11].

On the basis of crystallographic aspects, the ferrites are classified into three Major types.

1) Spinel

2) inverse

3) Hexagonal.

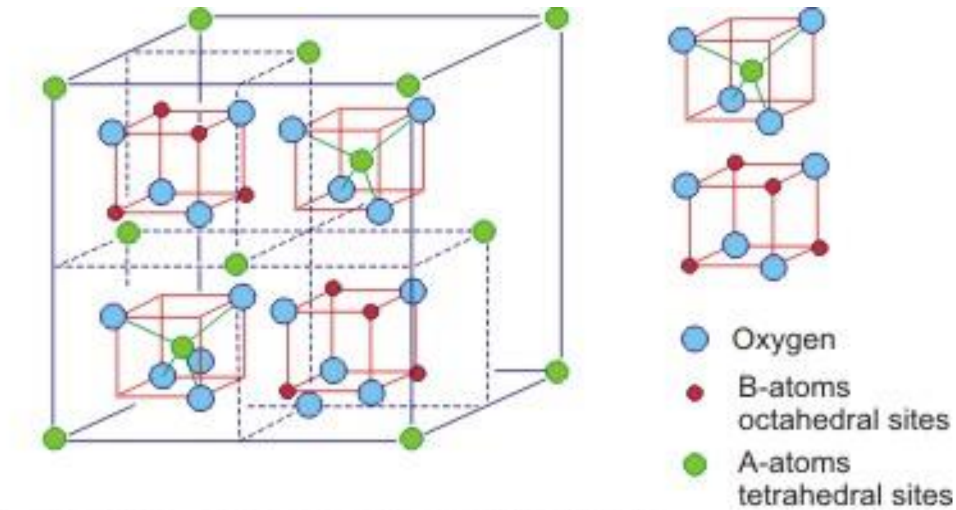
All of them are cubic in structure [12].

Ferrites are classified on the basis of arrangement of metal cations with giving different crystal structure.

Spinel ferrite:

Spinel ferrites are defined by the general formula AB_2O_4 having closed cubic crystal. Here A is any divalent ion that occupy the octahedral site in the crystal and B is trivalent cation that occupy octahedral sites. $MgAl_2O_4$ is the naturally exist spinel ferrite that was determined by Bragg [13] this is practically stable, if Al can be replaced by Fe it becomes one of the important example of spinel ferrites i.e. Fe_3O_4 called magnetite. The position of oxygen is FCC i.e. face centered cubic. which have two types of interstitial sites 64 tetrahedral and 32 octahedral sites only one –eighth of tetrahedral sites are occupied and half of tetrahedral sites are occupied by cations.

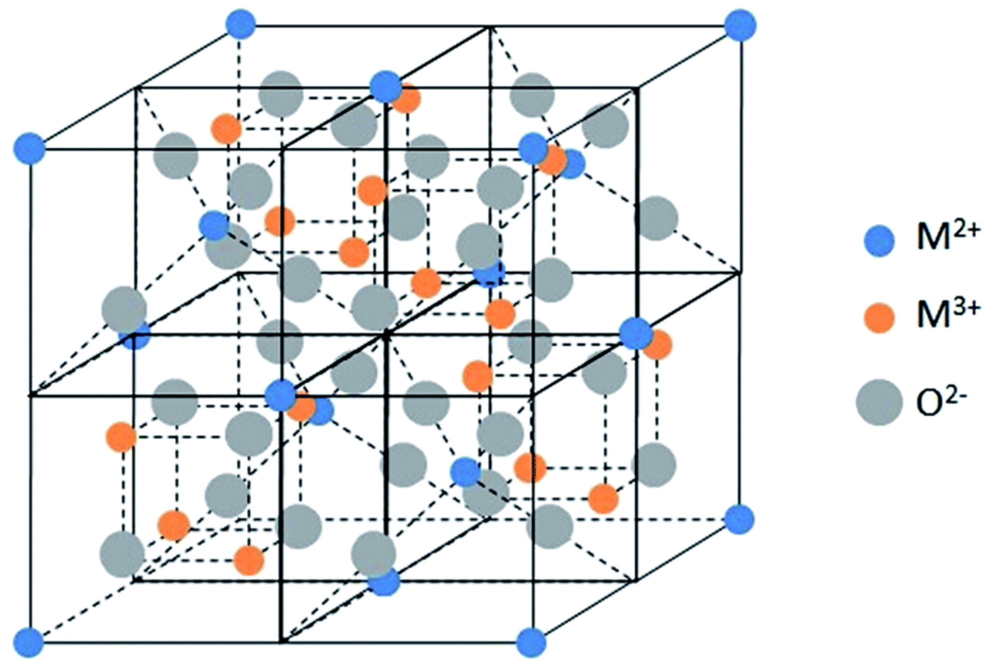
In $MgAl_2O_4$, Al and Mg cations occupy the octahedral and tetrahedral sites hence such type of cations arrangement is called spinel ferrites. Layers of oxygen ions contain 64 tetrahedral sites and 32 octahedral sites to provide the electrical neutrality of the lattice by 8 units to tetrahedral site and 16 units by octahedral sites, these sites are occupied by divalent or trivalent ions. Hence unit cell having formula AB_2O_4 [14].



AB_2O_4 spinel The red cubes are also contained in the back half of the unit cell.

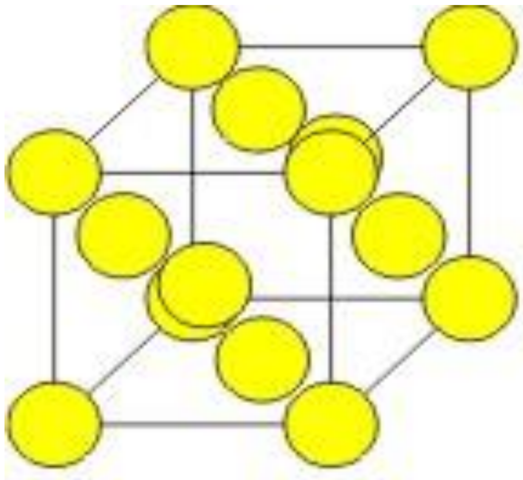
Inverse spinel:

Inverse spinel ferrites are defined as a alternative arrangement in which half of the tetrahedral sites are covered by the trivalent ion and half of octahedral sites are covered by divalent ions and rest of cations are randomly distributed on the octahedral sites. Example of inverse ferrites are $B(AB)O_4$ in which divalent cations such as Ni occupies the octahedral sites and half of Fe are at tetrahedral position.

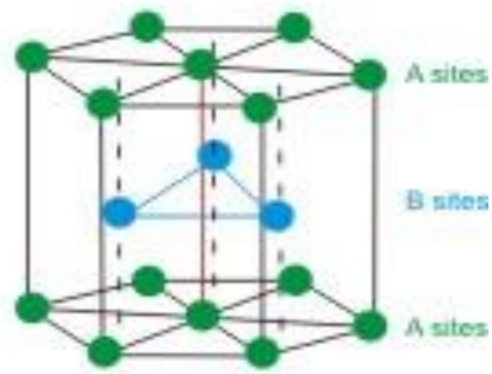


Hexagonal ferrite:

Hexagonal ferrites are the first discovered by the Jonker, Win and Barun in 1956. Hexagonal ferrites are also called hexa ferrites, they are magnetic oxides of iron with hexagonal structure having formula $MFe_{12}O_{19}$, where M is an element such as Barium, Lead, Strontium, in these ferrites oxygen ions have closed packed hexagonal structure. Hexagonal ferrites are used as a permanent magnet because they have high coercivity value and they can be used at high frequency. The hexagonal ferrite lattice is almost similar to spinal ferrite structure with closely packed oxygen ions but the only difference is that in hexagonal ferrites there are also metal ions at few layers with same ionic radii as oxygen ions. Hexagonal ferrites have ions with large size than garnet ferrite and are formed by the replacement of oxygen ions with barium, strontium and lead.



FCC/CCP Structure



HCP Structure

Fig:3(hexagonal Ferrite)

LITERATURE REVIEW

Manish et.al prepared the ferrites by sol gel method for frequency dependent dielectric measurement of Cd doped ferrite [15]. The dielectric properties and electrical conductivity depend upon the preparation and doping of cations. Binu et al. synthesized the Gd doped Cd ferrites and characterized by the XRD, Scanning electron Microscopy and Trans Electron Microscopy, which confirms the structure of spinel ferrites [16].

D. Makovee et al. synthesized a nano ferrite of zinc-Nikle bulk material i.e. characterized by the large affinity of zinc for the tetrahedral lattice sites also closely defined by stoichiomety[17]. M. Niyaifar are synthesized mixed ferrites of (Cd-Mn) by co- precipitation method and use high milling to reduce the size of manganese ferrite to nano range [18].

R. Raeisi Shahraki et al. synthesized zinc ferrite nanoparticles without successive calcinations, the nanoparticles are prepared at 20^0C. Superparamagnetic behaviour was shown by the nanoparticles formed at room temperature [19]. P.M. Prithviraj et al. synthesized Nano crystalline particles of Zinc ferrite by self-propagating low-Temperature combustion method [20]. Prita Pant et.al synthesized a series of single-phase $Ni_{(1-x)}Zn_{(x)}Fe_2O_4$ here (X=0.20,0.35,0.50,0.60) with in the average range of nano particles 35nm by the use of oxalate precursor method [21]. Zhinfeng et al. prepared a nano-crystalline Ni-Zn ferrite powders by refluxing method with narrow particle size distribution [22]. A. Chatterjee et.al prepared $Ni_{0.5}Zn_{0.5}Fe_2O_4$ crysatls with dimensions varying from 13-35nm taking silica-gel matrix as a starting material and sol-gel method was used for their preparation [23]. $(Ni_{0.5} Zn_{0.5} Fe_2O_4)x/Sio_2$ nano compositions are prepared by using tetraethylorthosilicates (TEoS) and metallic nitrates as starting materials by Sol-Gel Method and proceeding by anneling at temperature of 1273K for 1 hr [24]. A.E. Baranchikov et al. prepared Fe_2O_3 by thermal decomposition of analytical-grade $Fe(No_3)_3$ on a flame and annealed isothermally at 800^oC for 3 hr [25]. Behrrooz et al. synthesized hydroxyapatite-encapsulated Ni 0.5 Zn 0.5 Fe204 nanocrystallite as a catalyst for yhe synthesis of pyrazolo [1,2-b] phthalazine-5,10-diones [26]. Gouli fan et al. synthesized nano crystalline zinc ferrites ($ZnFe_2O_4$) with variation in their crystalline sizes, the process is used them to synthesis zinc ferrite arecolloid mill and hydrothermal technique [27]. Majid et al. prepared $Y_3Fe_5O_{12}$ nano particles which fall under garnet category, but they prepare the same ferrite by different methods which includes sol-gel method, self-combustion method and MCMO method [28]. Lin Zhuang et al.prepared temperature sensitive ferrofluides composed of $Mn_{(1-x)}Zn_{(x)}Fe_2O_4$ nano-particles by a modified hydrothermal process [29]. Kondal et al. synthesized a varity of spinel ferrites MFe_2O_4; here (M=Ni,Cu,Zn) i.e. in composition $Ni_{0.5}Cu_{0.5}Fe_20_4$, $Ni_{0.5}Zn_{0.5}Fe_2O_4$, $Ni_{0.5}Cu_{0.25}Fe_2O_4$ [30]. Milutinovin et al. prepared $NiFe_2O_4$ and $MnFe_2O_4$ by soft mechano chemical method by taking $Ni(OH)_2$ and alpha-Fe_2O_3 as reactants and $Mn(OH)_2$ or alpha-Fe_2O_3 are starting materials reapectively [31]. Kamelllia et al. synthesized one of the soft magnetic materials i.e. nickel ferrite ($NiFe_2O_4$) by hydrothermal method and is investigated for the inhibition of surfactants on particles growth [32]. Sajal et al. synthesized a Y-type hexagonal ferrite by conventional solid state reaction method [33]. Vishal et al. prepared nanoparticles of cobalt doped nikle ferrite $Ni_{(1-x)}Co_{(x)}Fe_2O_4$:here (x=0.5) by heat trearment method [34]. Poh et al. synthesized cubic structured Ni-Zn ferrite nanoparticles ($Ni_{0.25}$ $Zn_{0.75}Fe_2O_4$) by the thermal method [35].M.Siva kumar et al. prepared cobalt ferrite i.e. ($CoFe_2O_4$) nanoparticles by sol-gel method using nitrates, polyvinyl alcohol and citric acid [36]. G. Thirupathi et al. deliberated the magnetic behaviour of zinc ferrite nanoparticles ($ZnFe_2O_4$) formed by chemical co-precipitation method. The crystallite size was guarded by the pH of the solution and it was observed that the particles having crystallite size = 2nm shows paramagnetic behaviour while the particles having size greater than 2nm reveal superpar amagnetic property [37]. D.Gherca were synthesized nanocrystalline spinel ferrite MFe_2O_4 (M= Co, Mn, Ni) by a modified co-precipitation method using iron(III) chloride, Metal(II) chloride and canon oil solution i.e. used to prevent agglomeratration of nanoparticles [38]. M.Hessien et al. prepared nano-crystalline strontium hexaferrite ($SrFe_{12}O_{19}$) by using co-precipitation route [39]. R Galindo synthesized Nikle nano ferrites by four different synthetic wet routes a) Sono chemistry (SC), Electrochemistry(E), Co-precipitation(CP), and sonoelectro chemistry(SE) and studied the variation in the synthesis method that how leads to change in structural and magnetic properties [40]. A.E. Barachikov synthesiszed zinc ferrite by sono chemical synthesis and investigated for the evolution of defect structure of Fe_2O_3 and $ZnFe_2O_4$[41]. Ri-Chan synthesized magnetic nano-particles $Co_{(x)}Ni_{(1-x)}$Fe2O4,(X=0,0.3,0.7,0.9,1.0) by colloide mill and hydrothermal technique [42]. Rahul et al. synthesized a W-type barium hexaferrite $BaNi_2Fe_{16}O_{27}$ by using sol-gel method by taking citrate as a precursor followed by gel-to-nanocrystalline conversion; collectively the method is called Low-temperature Combustion method [43]. A.M. Huizar prepared $Sm_{(1-x)}Ca_{(x)}$FeO3(0.1<x<0.5) a compound by sol-gel based pechini method at low temperature [44]. Leena et al. prepared a series of nanoferrites of Nikle having formula $Ni_{(1-x)}Cu_{(x)}$Fe2O4 ;(0<x<0.9) by mechno chemical technique, in this series they studied the influence of copper substitution in nano ferrite, the spinel structure and lattice constants was investigated by Cohen Least Square fit Model [45]. M.A. Ali et al prepared the Ni-Zn ferrite with Sn-substitution by Standard double sintering technique taking oxide nanoparticles as a starting materials the formula for the compound is Ni0.6-x/2Zn0.4-x/2SnxFe2O4 ($0.0 \leq x \leq 0.30$) [46]. A.V.Knyazev et al. synthesized Ni-Zn and Ni-Zn-Co ferrite by solid state reaction synthesis with cobalt as a ceremonial composition, the chemical formula for the compound is $Ni_{0.5}Zn_{0.5}Fe_2O_4$ and Ni0.5Zn0.3Co0.2Fe2O4 [47]. M.K. Anupama et al. synthesized a mixed nanocrstalline ferrite of Ni and Zn by Low temperature self-combustion oxylyldehydrazide (ODH) as a starting fuel, the synthesized compound having formula $Ni_{1-x}Zn_x$ Fe2O4 (x= 0.0, 0.2, 0.4, 0.6, 0.8 and 1) [48]. Raul et al. synthesized NI-Zn ferrite by polyol method proceded by spark plasma sintering at 600^{o}c for 10 min to convert them into solid fine grain [49]. C.Y. Tsal et al. prepared green compact of ferrite material by taking commercial ferrites materials as starting materials, the synthesized compound having formula ($Ni_{1-x-y}Zn_xCu_y$) [50]. Reda et al. synthesized nanocrystalline potassium zinc hexacyanoferrate, the compound is synthesiszed for removal of cesium from low-level radioactive waste [51]. Cs3[Fe(L)6].xH2O (L = formate, acetate etc) were prepared by adding appreciate quantities of aqouse solution of ferrite nitrate ,cesium carboxylate and carboxylic acid [52].

Methods of Synthesis of Ferrites

Sol-Gel Auto Combustion Method: Huge developments and trends of sol-gel auto combustion methods for spinel ferrite nano material synthesis are briefly explained and critically analyzed. The analysis of various steps of reaction helps in better understanding of synthesis technique. The main focus was on the variety of particle size; for this purpose, correlation between oxygen balance and combustion process, chemical additives, heating mechanisms was established. [53].

This method is exothermic reaction and self-sustaining thermally induced anionic redox reaction of xerogel i.e. obtained from aqueous solution containing desired metal salts and organic complexant [54]. Properties between salt and complexant are calculated according to the valences of the reacting elements in order to maintain the relation of oxidizer/reductant equal to 1 [55]. The use of nitrate salts are in Sol-Gel auto combustion method for the synthesis of nano sized ferrites. These nitrate salts are act as precursors due to their water-soluble nature at low temperature. Metal nitrates and complexant are directly added together by continues stirring and heating (no use of water). Since metal nitrates are hygroscopic in nature so they easily absorbs moisture and becomes like slurry at this moment sol-Gel Auto-Combustion method is called flash-Combustion method[56].

The yield of product is less in Sol-Gel Auto-Combustion method because rapid evolution of large volume of gasses that leads to great mass loss during the xero gel combustion and the formation of ferrite nano powders. Evolution of gasses limits the occurrence of agglomeration [25-26]. Flame temperature during the combustion vary from 600^0C-1350^0C [57].

Fly Ash Method: O.A Fouad.et.al worked on catalytic oxidation of CO over synthesized nickel ferrites prepared from fly ash method has been investigated, these investigated studies are shown that pure crystalline forms of ferrites are obtained by thermal treatment of the precursors at temperature>800^0C for 120 min under pH considerations from 7 to 12.

At low temperature, i.e. $50<T<800^0$C impure low crystalline $NiFe_2O_4$ phase formed. The main source of impurities are FeO (OH),$Fe_2O_3.H_2O$ phase.

At high temperature of 1200^0C for 120 min the higher magnetization is obtained for a precursor precipitation at pH=10[58].

Facile Bubble Assisted method: Pure porous hollow Zn ferrites($ZnFe_2O_4$) micro spheres are synthesized by a facile bubble assisted method in the presence of ammonium acetate as an anode material in lithium ion battery [59]. The shape, size, morphology of Zn ferrites are investigated by X-ray diffraction, Scanning electron microscopy and Trans-mission electron microscopy[60].

The techniques used for the synthesis of mixed ferrites are also Ball Milling and Co-Precipitation [61] $Mn_{(x)}Ni_{(1-x)}Fe_2O_4$ nanocrystalline mixed ferrites are also synthesized by equilibrated low entropy route[62].Also Hydrothermal method is used for the synthesis of mixed ferrites of Cobalt-Nickel nanoparticles $Co_{(x)}Ni_{(1-x)}Fe_2O_4$[63]. Synthesis of Zinc-Cobalt mixed ferrites nanoparticles is through hydrothermal method and studied their magnetic and structural properties[64]. Singh.et.al used solution combustion method for the synthesis of Li-Sr-Zn substitution on magnetic electrical and structural properties of the ferrites[65]. Gupta.et.al investigated physical-chemical properties of pure and Zn doped Cd ferrites prepared by Pechini Sol-gel method using citric acid-glycol and complexing agents[66]. carbon combustion synthesis method is also applied to get fast and energy efficient fabrication of crystalline of barium hexaferrite nanoparticles, in this method approximate particle sizes 50-100nm.Since this is a exothermic reaction which involves the oxidation of carbon nanoparticles those average size of 5nm generates a self-propagating thermal wave with temperature up to 1000^0C,here thermal front plays a important role for the synthesis. The thermal front rapidly propagates through the mixture of solid reactants converting them to hexagonal barium ferrites, but carbon is get evolved from the reaction mixture as a gaseous CO_2.Activation energy of carbon combustion synthesis varies as composition and metal changes[67].

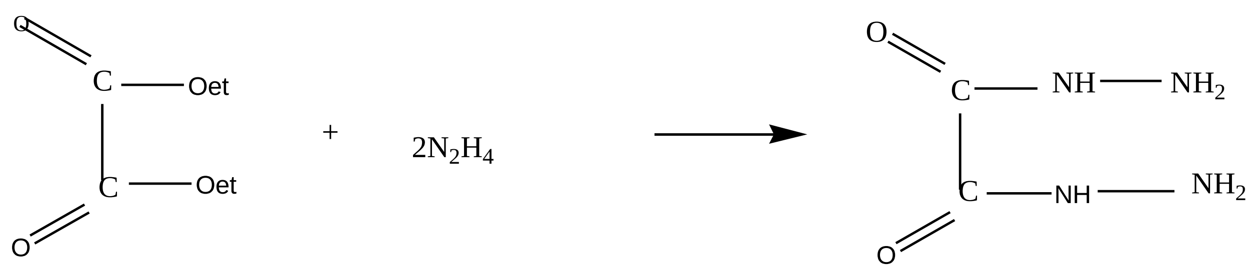
EXPERIMENTAL WORK

For the preparation of Nano-sized transition metal ferrites particles the first step is the synthesis of ODH (oxalyldihydrazine), which acts as a fuel for combustion synthesis.

Synthesis of ODH

1 mole addition of diethyl oxalate to 2 mole of hydrazine hydrate at 4^0C, since this is highly exothermic reaction so the addition of diethyl oxalate should be drop wise in the hydrazine hydrate and proper stirring is maintained during the addition. White colour precipitates are formed which are then washed with cold water.

REACTIONS INCLUDED:



Diethyloxalate Hydrazinehydrate Oxaloyldihydrazine

Synthesis of ferrites:

Ferrite materials with composition $Ni0._{75-x}Cs_{0.25}Zn_xFe_2O_4$ were synthesized by using solution combustion method. Series of ferrite compositions were prepared where x varies from 0 to 0.5 in steps of 0.1.

Step 1: Stoichiometric aqueous solutions were prepared form respective metal nitrates in minimum amount of distilled water.

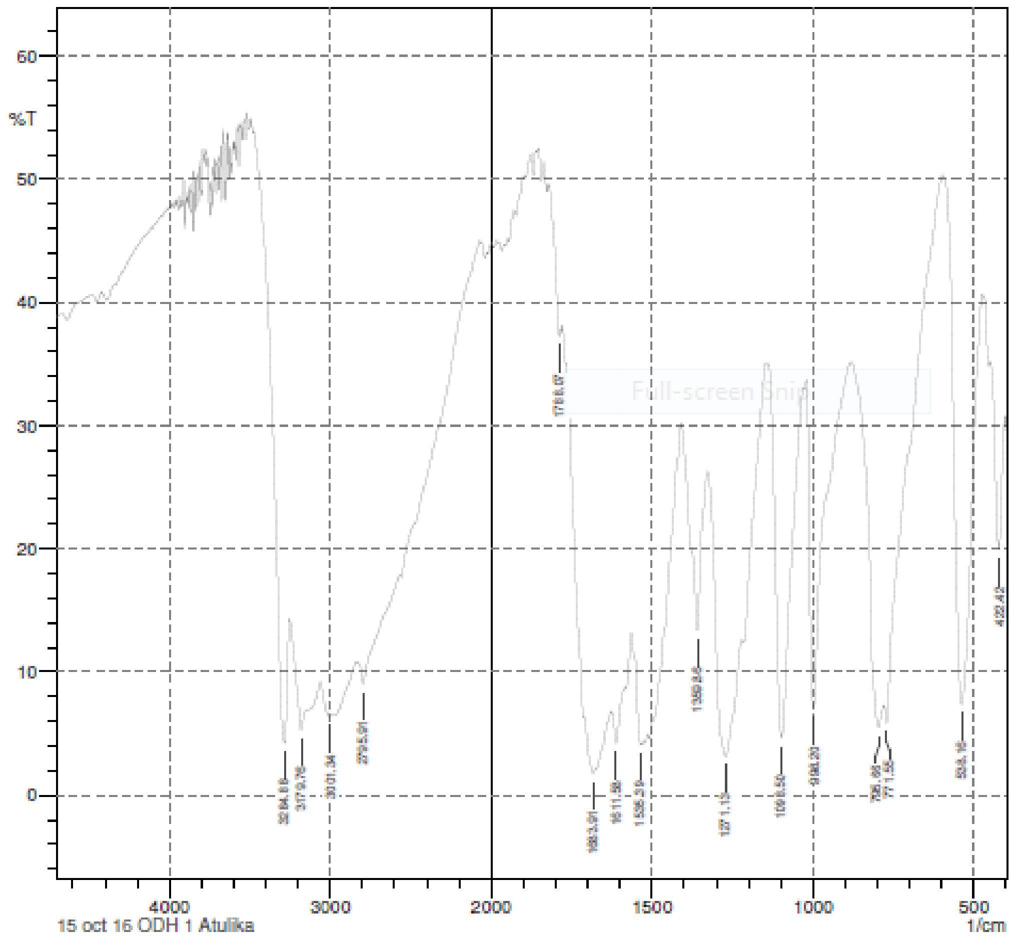
Step 2: Different solutions were mixed in a single beaker.

Step 3: ODH paste was added slowly into the reaction mixture as reaction is highly exothermic.

Step 4: After complete addition of ODH, reaction mixture was concentrated on water bath.

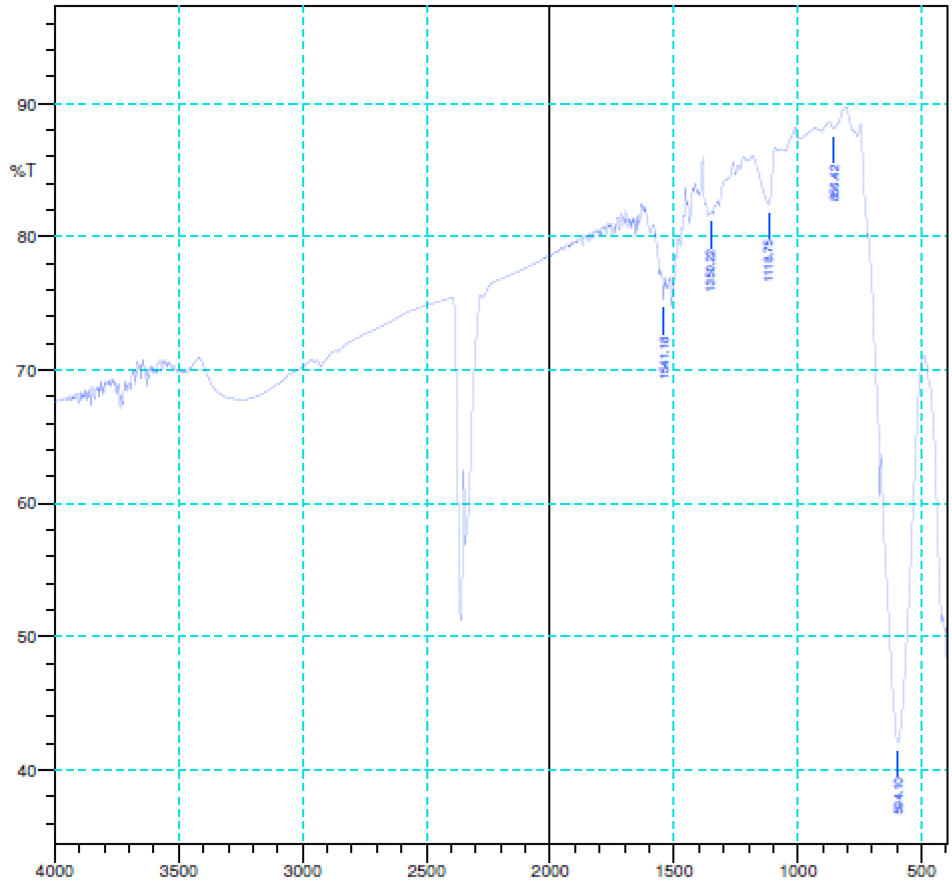
Step 5: The concentrate was then subjected to heating in muffle furnace up to 650^0C to obtain the final ferrite powder.

Step 6: The final powder product was then subjected to characterizations.

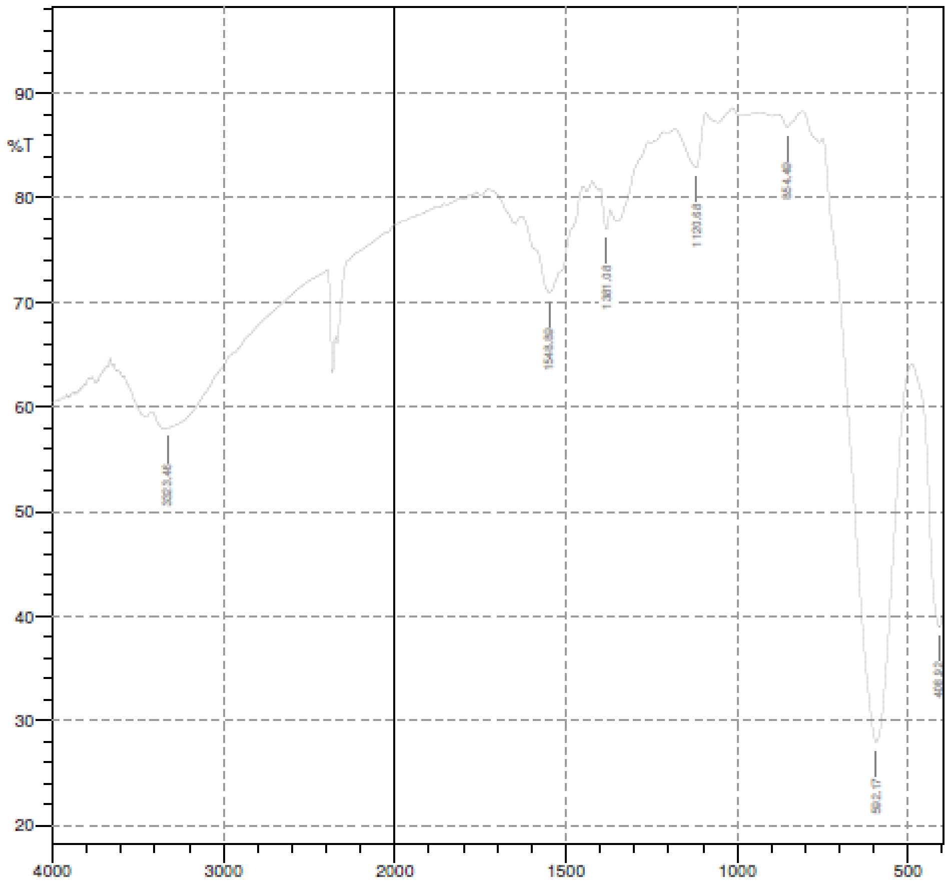


FT-IR Spectum of Oxalyl dihydrazide (ODH)

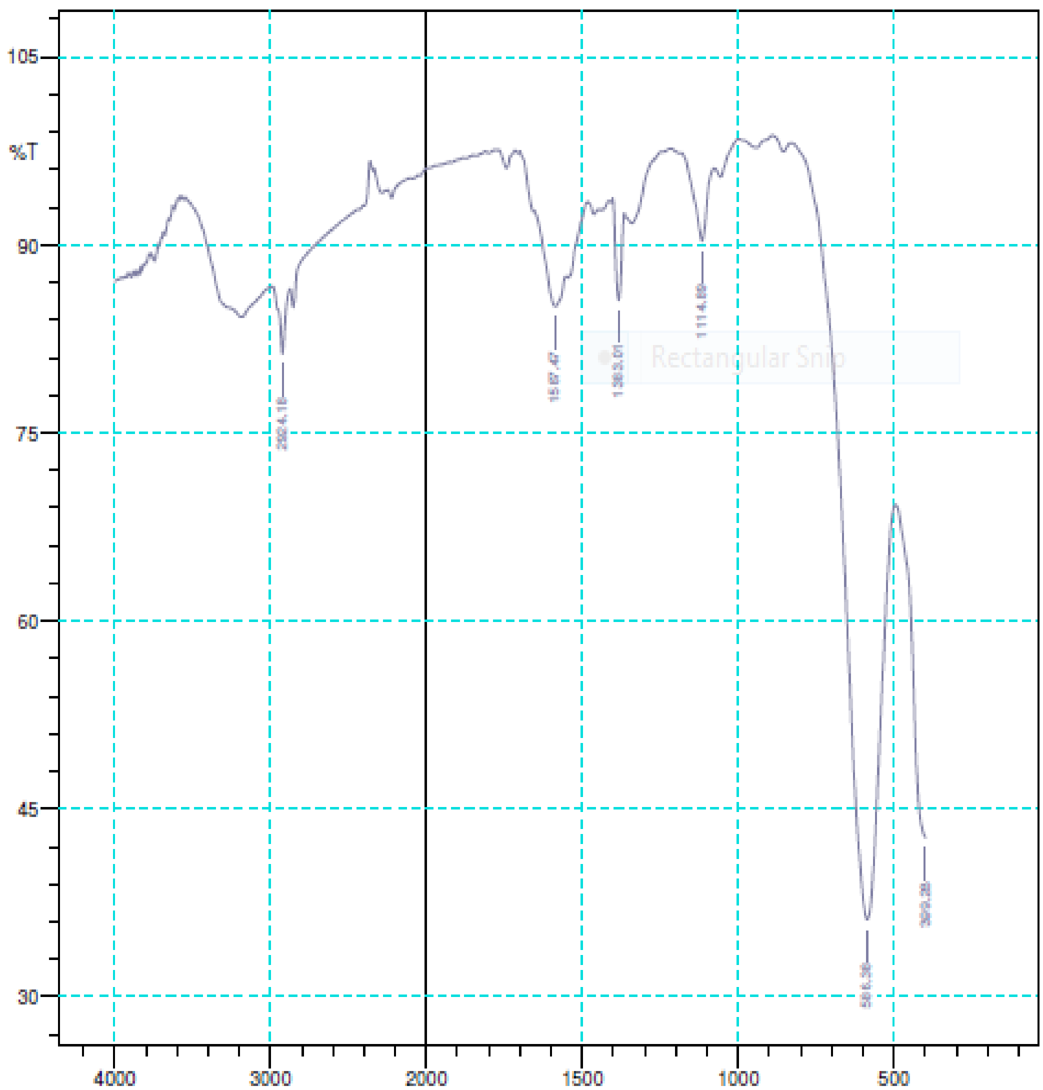
FTIR spectra of ferrite samples with varying composition show two absorption bands in the range 600 cm^{-1} to 400 cm^{-1} for the tetrahedral (at higher frequency) and the octahedral (at lower frequency) sites respectively. These two absorption bands indicate the formation of spinel structure. Absorption band around 600 cm^{-1} is due to the metal-oxygen intrinsic vibrations for tetrahedral complexes and band below 450 cm^{-1} is due to the octahedral metal-oxygen intrinsic vibrations. This difference in the frequencies of distinctive vibrations for tetrahedral and octahedral sites arises because of different bond lengths for the metal-oxygen ions in corresponding sites.



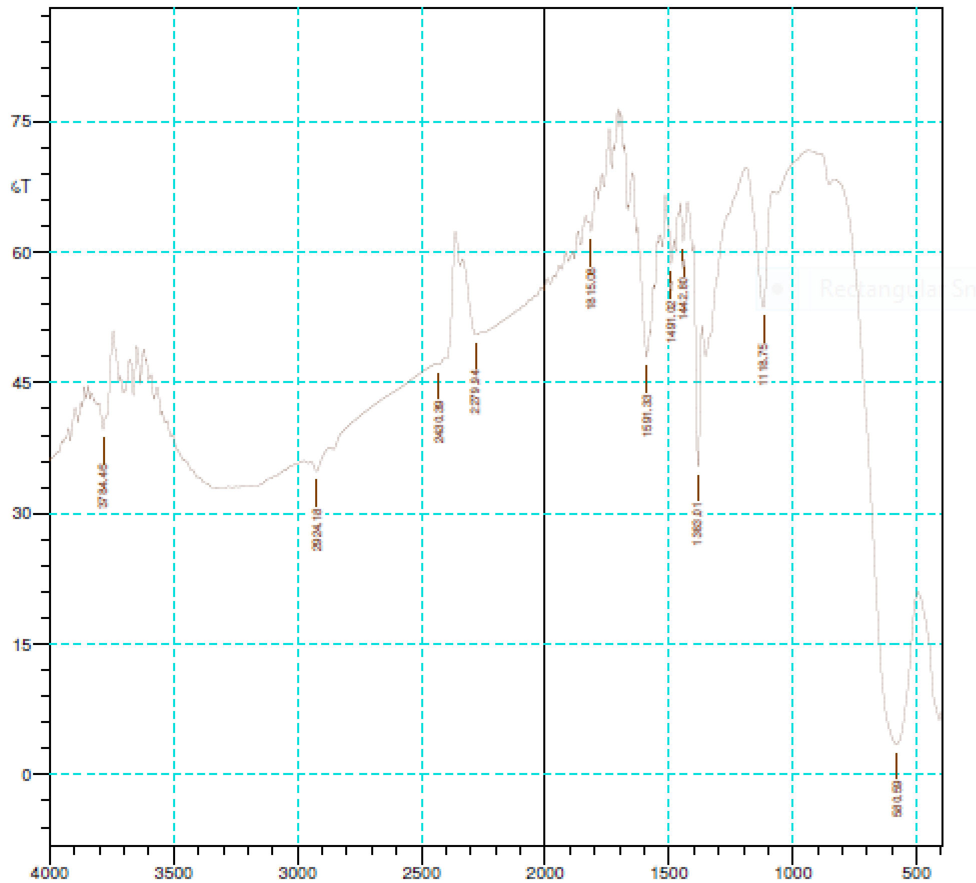
FT-IR spectrum of Ni0.$_{75-x}$$Cs_{0.25}Zn_xFe_2O_4$, where X=0



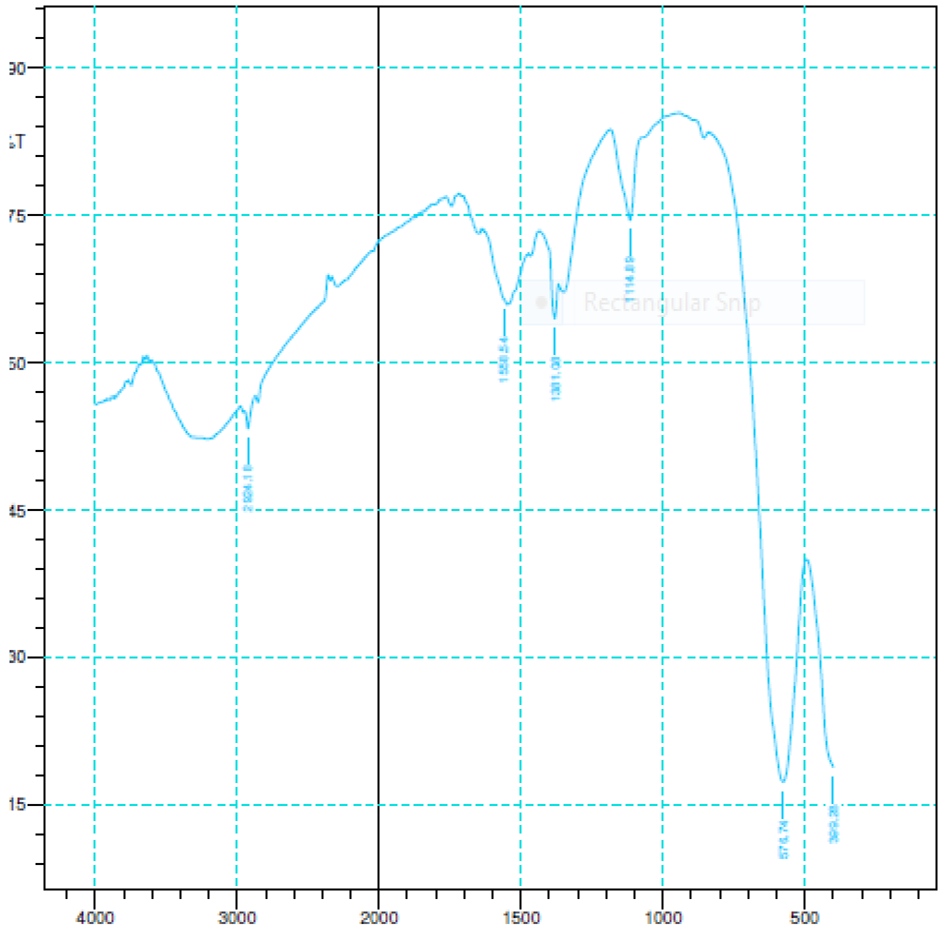
FT-IR spectrum of $Ni0._{75-x}Cs_{0.25}Zn_xFe_2O_4$, where X=0.1



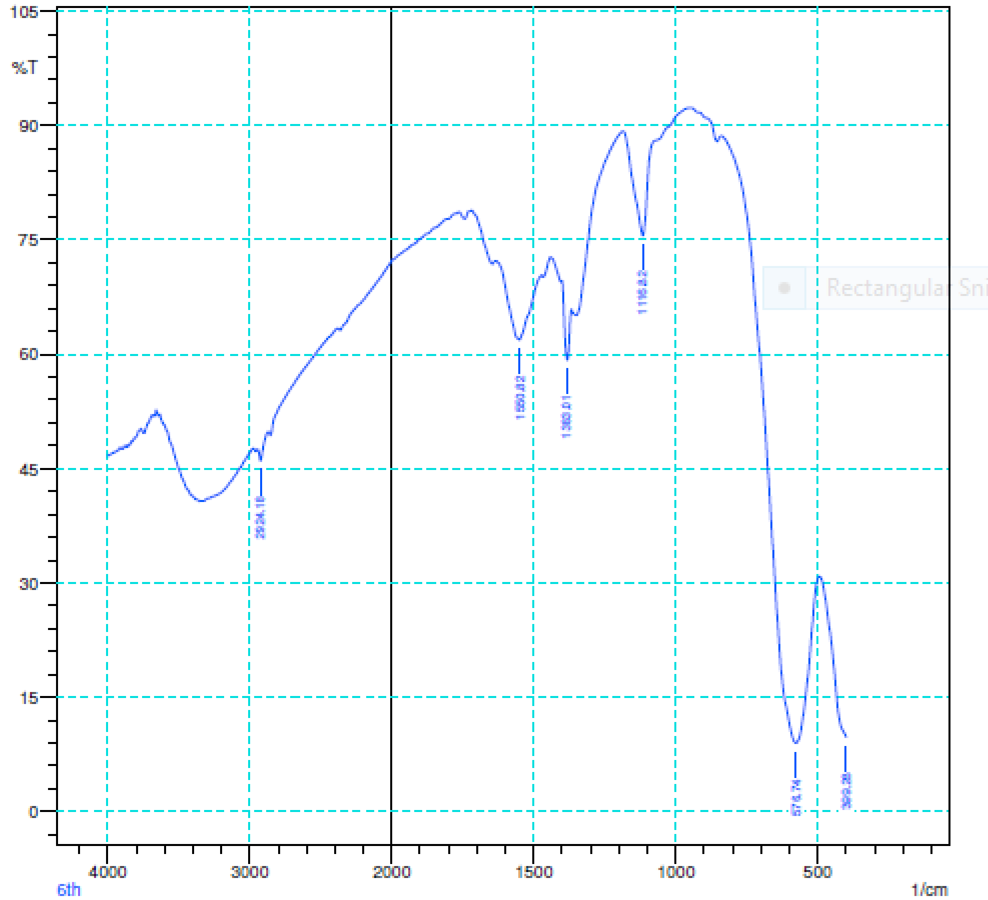
FT-IR spectrum of $Ni0._{75-x}Cs_{0.25}Zn_xFe_2O_4$, where X=0.2



FT-IR spectrum of $Ni0._{75-x}Cs_{0.25}Zn_xFe_2O_4$, where X=0.3



FT-IR spectrum of Ni0.$_{75-x}$Cs$_{0.25}$Zn$_x$Fe$_2$O$_4$, where X=0.4



FT-IR spectrum of Ni0.$_{75-x}$Cs$_{0.25}$Zn$_x$Fe$_2$O$_4$, where X=0.5

CONCLUSIONS

Solution combustion method has many advantages over conventional ceramic methods as follows;

1. ODH acts as a fuel for combustion synthesis.
2. Ferrite materials are synthesized at lower temperature.
3. Ferrite materials are synthesized in shorter time.
4. Combustion process dissipates heat during combustion which prevents the aggregation of particles and nano particles can be synthesized.
5. Single phase spinel ferrites are synthesized due to stoichiometric mixing of reactants.

Further, magnetic studies of these ferrite materials can be explored to check their behavior as hard or soft ferrites regarding their applications in potential fields.

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