SYNTHESIS AND CHARACTERIZATION OF CESIUM FERRITE AND ITS APPLICATION IN ADSORPTION OF DYES

Dissertation Project Report-1submitted to

Lovely Professional University, India

For the partial fulfilment of the award of degree

Of

Masters of Science in Chemistry (Honours)

By

Nisha

(11610667)

Under the guidance of

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November 2017

DECLARATION

I hereby declare that the dissertation report entitled**, "Synthesis and Characterization of cesium ferrite and its application in adsorption of dyes"** submitted for the M.Sc. Chemistry (Hons.) degree is entirely my original work and all ideas and references have been duly acknowledged. It does not contain any work for the award of any other degree or diploma at any university.

Date: 30 November, 2017

Nisha

Reg. No. 11610667

CERTIFICATE

This is to certify that Nisha has completed the dissertation-1 report entitled "Synthesis and Characterization of Cesium Ferrite and its application in adsorption of dyes"**,** under my guidance and supervision. To the best of my knowledge, the present work is the result of her original investigation and study.

Date: 30 November, 2017

Dr. Harmanjit Singh Dosanjh

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I am highly indebted to “Dr. Harmanjit Singh Dosanjh” for his guidance and constant supervision as well as for providing necessary information regarding the project and also for his support where I need the most.

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INTRODUCTION

FERRITES:

Ferrites are generally the magnetic materials, which have the combined magnetic and electrical properties. Iron and metal oxides are main constituents of ferrites. The importance of ferrites has been known for mankind for many centuries. The study of ferrite has been started in the year of 1930. Many researchers and scientists have studied the structure and the magnetic properties of ferrites and their uses. (1)

PROPERTIES:

FERRITES generally contain many properties:-

- Ferrites material is insulating magnetic oxides and possess high electrical resistivity.
- High saturation magnetization.
- Low eddy current.
- High permeability and high permittivity.
- Thus, very rare materials exhibit such wide range of properties, due to such variety of ferrites material, these materials find applications in various fields. (2)

USES:

They are used in transformer cores.

- Antenna rod.
- memory chips
- permanent magnet
- microwave and computer application (3)

WHY FERRITES:

As ferrites have variety of property and uses. The first factor is dielectric property of ferrites. Exhibiting dielectric property mean when electromagnetic wave passes through the ferrites, they do not conduct the electricity, while the other transition metal may conduct electricity. Another factor is the Porosity. (4)

TYPES OF FERRITE

Basically, ferrites are of generally of three types:

1. Spinel

2. Garnet

3. Magneto-plumbite

All these type of ferrite have their identity.

- Spinel ferrites are represented by chemical formula MFe_2O_4 (where M is divalent cation such as Co, Zn, Ni, Cd)
- Garnet have the chemical formula $R_3Fe_5O_{12}$ (Where R is the rare earth element like Dy, Gd, La etc.)
- Magneto plumbite have the chemical formula $MFe_{12}O_9$ (where M =Ba, Sr, Ca etc)

1. SPINEL FERRITE:

These ferrites are also called as cubic ferrite. Spinel is the most widely used type of ferrite. The spinel structure of the ferrite as possessed by mineral spinel ($MgAl_2O_4$) was first determined by BRAG and NISHIKAWA in 1915. As spinel ferrite are generally represented by chemical formula MFe_2O_4.Thus, the unit cell of the spinel ferrite is Face centre cubic (FCC) with eight formula units per unit cell. Thus, it can be written as $M_8Fe_{16}O_{32}$. With this type of lattice, two types of interstitial position occur and these sites are occupied by the metallic positions. The total interstitial site in unit cell is 96 in which 64 is tetrahedral site and 32 is octahedral site. (5)

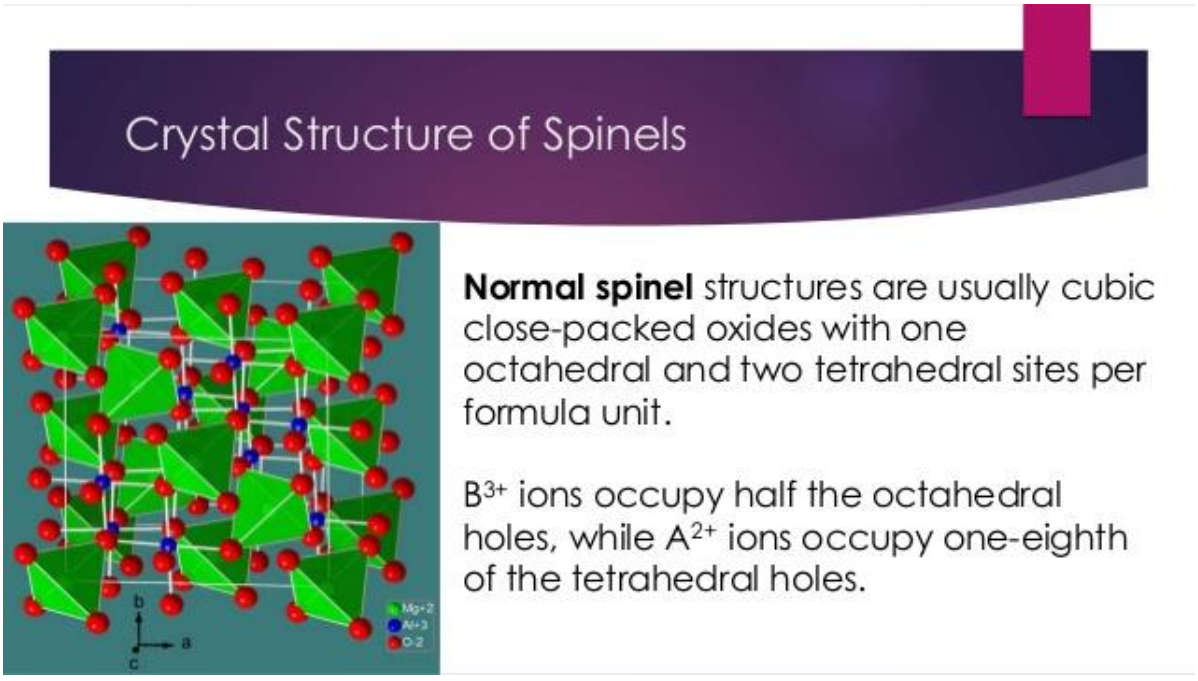
On the basis of distribution of cation on octahedral and tetrahedral site, spinel ferrites are categorized into three types:

a) Normal spinel

b) Inverse spinel

c) Intermediate spinel

a) NORMAL SPINEL:

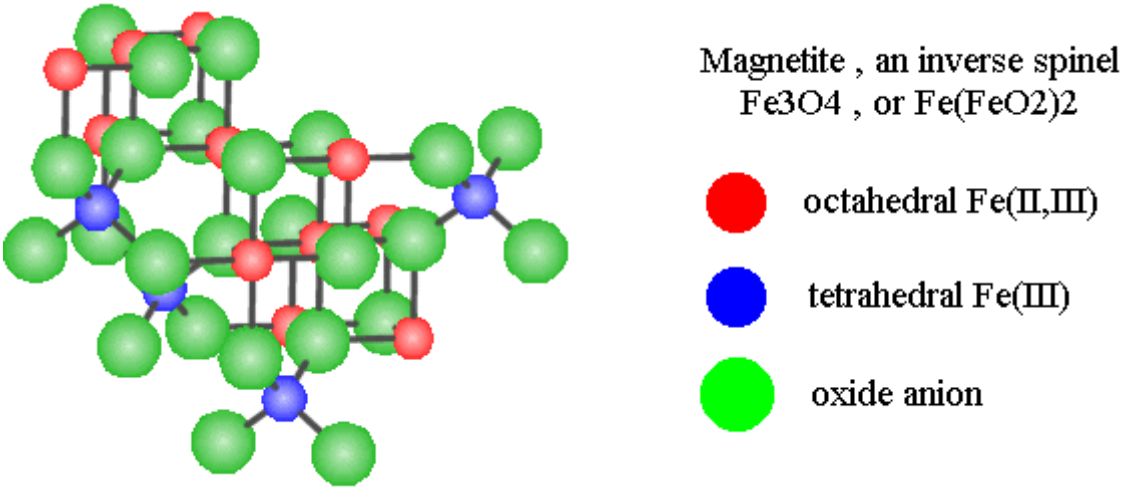


In this type, the one kind of cation occupies the octahedral site. For e.g.: $ZnFe_2O_4$. In this Zn occupies tetrahedral site and Fe occupies the octahedral site.

Structure (1a) (https://www.slideshare.net/shobana3/solid-state-43045558)

b) INVERSE SPINEL:

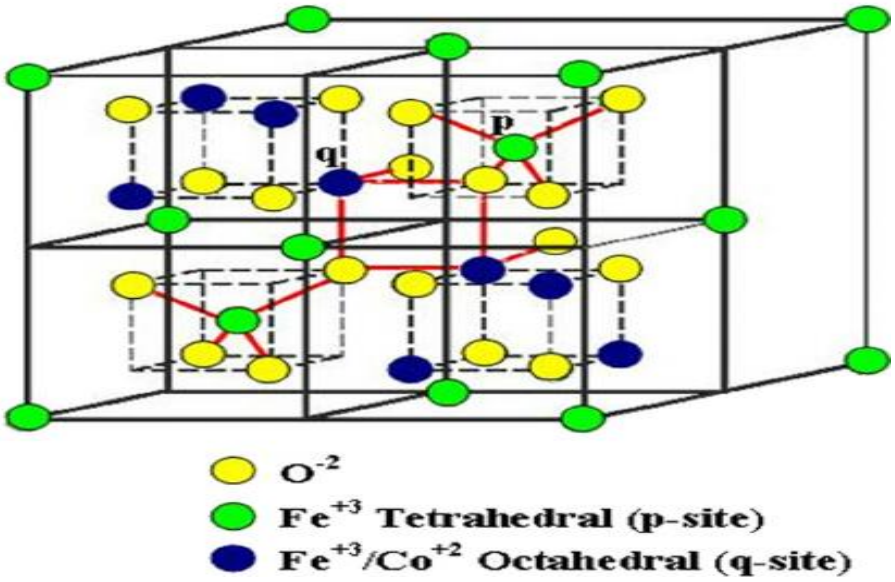
In inverses spinel, half of the trivalent ions occupy tetrahedral sites and the half octahedral sites, the remaining cation are randomly distributed among octahedral sites. For e.g. Fe_3O_4 (6)



Structure (2a) (http://www.pisanieprac.info/2017/inverse-spinel-structure.tech)

c) INTERMEDIATE SPINEL:

It is also called as mixed spinel which means that this spinel is intermediate between normal and inverse spinel structure. For e.g. $MgFe_2O_4$ or $MnFe_2O_4$. (7)



Structure (3a) (https://www.researchgate.net/figure/235926410_fig1_FIG-1-Color-online-Inverse-spinel-structure-showing-the-location-of-constituents)

2. MEGNETO –PLUMBITE FERRITE:

They are also called as hexagonal ferrites. Went, Rathenau Gorter and Van Oostershout (1952) and Jouker, Wijn and Braun (1956), are some of scientist or researchers who identify the type of ferrite. These including the chemical formula $MFe_{12}O_{19}$.In this type of ferrites, the oxygen ions have the closed packed hexagonal crystal structure. These types of ferrites are widely used in permanent magnets. (8)

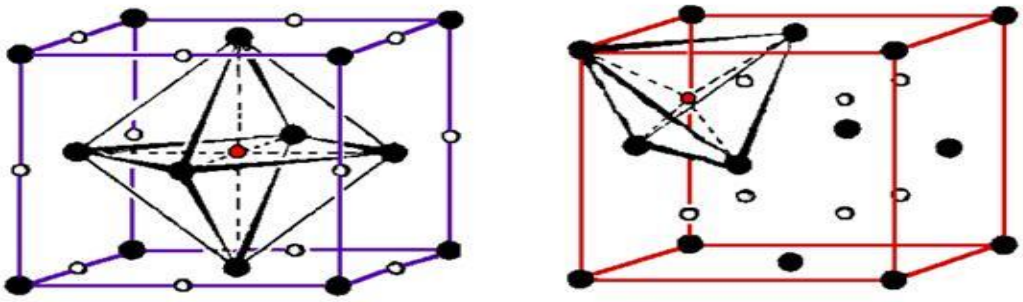
3. GARNET FERRITE:

There were many researchers and scientist in which one of Bartant and Forret who prepared $Y_3Fe_5O_{12}$ in 1956. In 1957 Gellor and Gilleo prepared the $Gd_3Fe_5O_{12}$ which is also ferromagnetic compound. General formula for garnet is $M_3Fe_5O_{12}$ where M is rare earth ion Gd, Y, Dy. Their cell shape is cubic. They are having the complex cubic structure but they have the importance in their application in memory structure.

Now we know that there are three types of ferrites i.e. spinel, magneto plumbite and garnet, and we can easily distinguish that by knowing the chemical formula and metal atom attached to iron. But in case of spinel, they are further classified into three type i.e. normal inverse and intermediate.

There are various factors that determining cation distribution include the size of the cation, valency of the cation, and the oxygen parameter of anion.

Large divalent ion tends to occupy the tetrahedral site as this is favoured by polarisation effects.



Structure (4a) (http://www.askiitians.com/forums/Physical-Chemistry/why-number-of-octahedral-voids-is-n-and-tetrahedra_143258.htm)

For e.g. consider A for divalent cation occupies tetrahedral site and B for trivalent cations occupies octahedral site. If A site ion have less valency and B site ion have the high valency then intermediate O_2^- ion will become polarised towards B sites. Thus, polarisation favours normal spinel configuration.

Also, the inverse structure has the lowest lattice energy for μ parameter ($\mu<0.379$) whereas the normal spinel structure has lowest energy ($\mu>0.379$).

Thus, $CoFe_2O_4$ have the inverse spinel structure and $ZnFe_2O_4$ have normal spinel structure.

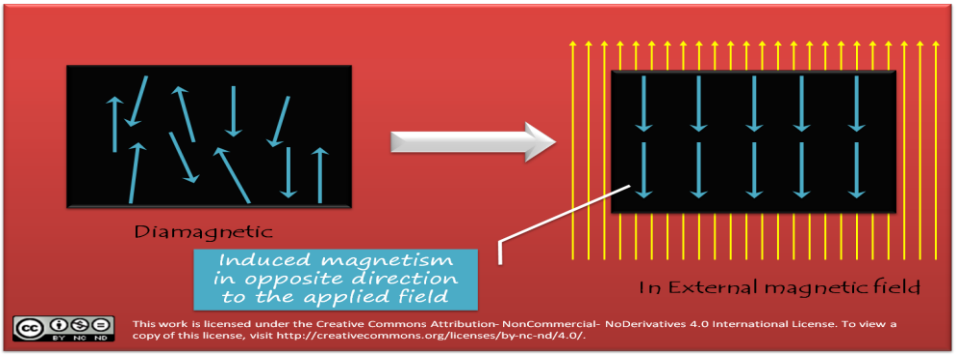
MAGNETIC PROPERTIES OF FERRITE

Magnetism comprises physical phenomena which involve magnetic fields and its effects upon materials. Magnetic fields can be set up on macroscopic scale by the electric currents or by the magnets. Thus, a material is magnetically characterised based on the way it can be magnetized.The types of magnetism are:

- Diamagnetism
- Paramagnetism
- Ferro-magnetism, anti-ferromagnetism and ferri-magnetism are considered as subclasses of ferromagnetism.

DIAMEGNETISM

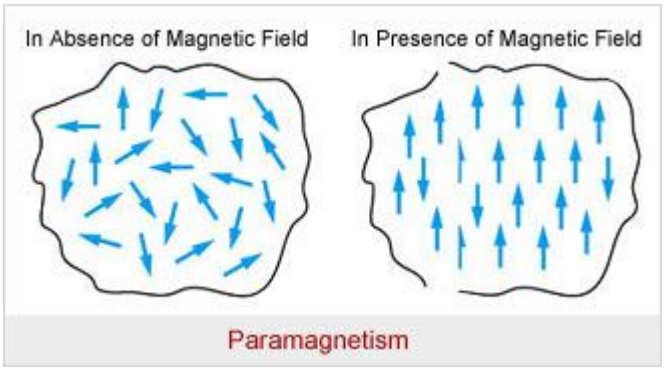
In the presence of magnetic field, when a diamagnetic substances is placed, they possess magnetic moment induced in them,which opposes the direction of the applied magnetic field. Thus, these types of substances are repelled by magnetic field. And permanent dipoles are absent in diamagnetic material.



Structure(5a)(Link:http://chemistrynotmystery.blogspot.in/2014/09/why-is-o2-paramagnetic-while-n2.html.)

PARAMEGNETISM

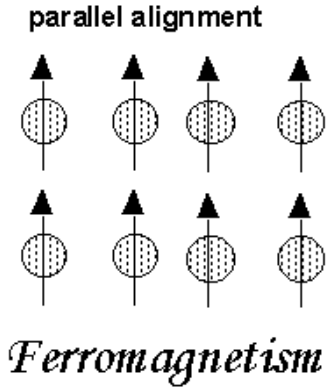
Paramagnetic materials result, when applied magnetic field lines up all existing magnetic moments of individual atoms or molecules making up that material. This results in an overall magnetic moment that adds to magnetic field. Paramagnetic structures mostly contain transition metals or rare earth materials that possess unpaired electrons.



Structure (6a) (http://elec-trical.blogspot.in/2012/06/types-of-magnetism.html)

FERROMEGNETISM

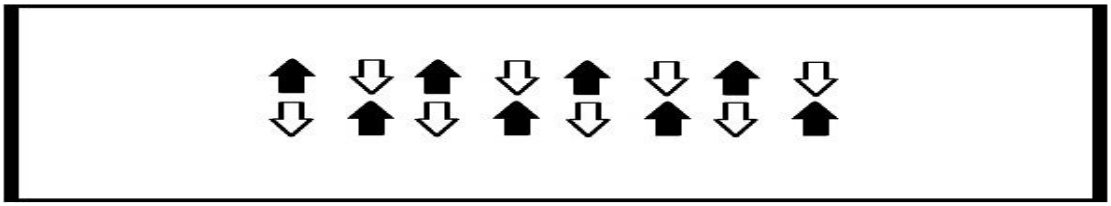
A ferromagnetic substance is one that retains the magnetic moment even when external magnetic field is lowered to zero. This effect is due to strong interaction between magnetic moments of individual atoms or electrons in a magnetic substance, that causes it to line up parallel to one another.



Structure(7a) (http://www.irm.umn.edu/hg2m/hg2m_b/hg2m_b.html)

ANTIFERROMEGNETISM

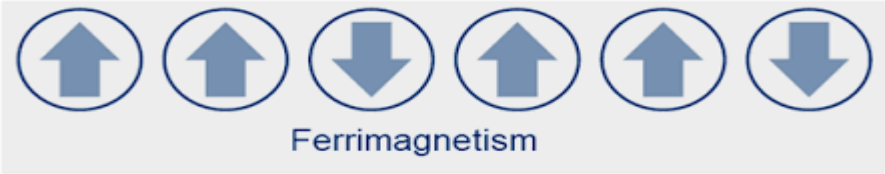
Antiferromegnetic substances in which the magnetic moments of atoms or the molecules, mostly related to the spins of electrons, align in regular pattern with neighbouring pointing in opposite directions.



Structure
(8a)https://eng.libretexts.org/Core/Materials_Science/Magnetic_Properties/Antiferromagnetism.

FERRIMEGNTISM

Ferromagnetism is generally involves in which the magnetic interaction between any two dipoles aligns anti parallel to each other. But since the magnitude of dipole are not equal. The cancellation of magnetic moments becomes incomplete resulting in net magnetisation in material. (9)

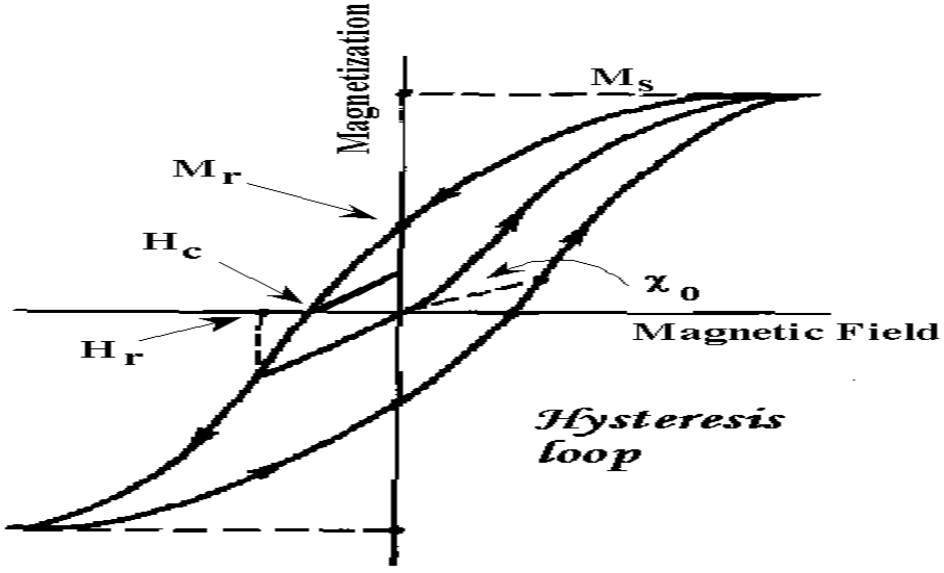


Structure (9a)

http://ncerthelp.com/text.php?contype=Exercise&class_id=12&sub_id=C&chapter_id=CH1&q_no=26

HYSTERISIS LOSS

The energy which is imparted or extended in reorienting domains from magnetized back to demagnetized state itself in a lag in response.This is known as hysteresis. (10)



Structure (10) (http://www.irm.umn.edu/hg2m/hg2m_b/hg2m_b.html)

The curve drawn for hysteresis is known as hysteresis loop.

METHOD OF SYNTHESIS OF FERRITE:
For the physio chemical properties, the synthesis of ferrite is an important factor. In general the synthesis of ferrites, there are many methods such as solid-state reaction, chemical co-

precipitation, sol-gel auto combustion, hydrothermal, microemulsion etc which can be employed for the synthesis of ferrites. Thus, the choosing the synthesis method is important to control the composition, structure, morphology and many of the properties of a prepared material. The brief discussion about synthesis methods are as follows.

1) SOLID STATE REACTION (CERAMIC METHOD):

The most common method used for the synthesis of ferrite is solid state reaction method in which we take ferrite material in bulk. In this method,we used the raw materials for the synthesis of ferrites are usually powders of Iron-III oxide and metal-II oxides such as CuO,NiO and ZnO, etc.In this method two important steps are calcinations and sintering.Firstly, we do the grinding and mixing for the homogenization of oxides to get the phase.In sintering process, the pellet of the material must be fired at maximum high temperature without melting. The reaction condition for this method is high temperatures (more than 1100^0C), where the reacting atoms can diffuse through the solid materials.The main disadvantage of this technique is that final materials are not pure and have additional phases.

2) WET CHEMICAL METHOD:

In wet chemical method,metal cations are coprecipitated from the common medium usually as chlorides, sulphates, nitrates, hydroxides,oxalates,carbonates and are subjected to heating at low temperature to yield final product. This method is used for preparation of polycrystalline samples, such as ferrite.This will involve three sub categories.

(a) CHEMICAL CO-PRECIPITATION METHOD:

This method generally involves the mixing of chlorides, sulphates in proper proportions in water (distilled). A 2M solution of NaOH was prepared as precipitant.Firstly the starting solution i.e mixing of chlorides and sulphates are added into the precipitant.In precipitation of all the hydroxides starting solution with pH-3 was added to solution of NaOH and to make suspension up to pH-11 containing dark intermediate precipitation. Then this suspension was heated at temperature 600^0C and oxygen gas was bubbled uniformly into suspension to stir it and to promote oxidation reaction until all the intermediate precipitant changed into dark brownish precipitate. The disadvantage of this method is. (11, 12)

1. Homogeneous nature of mixed precipitant
2. Synthesis ate low temperature
3. Control morphology of products

(b) MICRO EMULSION METHOD:

In this process, two chemicals are chosen in such a way that one of them is soluble in water and other is in the organic soluble only. Emulsion is made by mixing small volume of water in large volume of organic phase. As size of the water droplets are directly related to ratio of water to surfactant. Then after that surfactant molecules collect on surface of the water drop and stabilize the drop. This droplet is termed as reverse miscible. Since drop is small, only small amount of the reactants can squeeze into it. When drop reacts with another reactant, a tiny particle is formed. Thus,nanoparticle was synthesized and prevented from growing out of nano-regime. (13, 14)

(c) SOL-GEL AUTO COMBUSTION METHOD:

Sol gel auto combustion method is one of the best methods for the synthesis of ferrite. The raw materials that are used for the synthesis of ferrites are metal nitrates. In this method a highly exothermic redox reaction between nitrate and citrate ions takes place to produce the desired product. Nitrate ions act as burning oxidizers while citrate ions behave as reducing agents. The combustion method carried out in an air atmosphere. The two important roles of citric acid are: it is the fuel for the combustion reaction, and it forms stable complexes with the metal ions preventing precipitation of hydroxylated compounds The disadvantage of this technique is energy efficiency, low processing cost, relatively homogeneous mixing, low crystallization temperature, good stoichiometric control, and the production of fine crystalline particles in a relatively short processing time.

Advantages of the chemical methods:

1. The main advantage of the chemical methods is the mixing of reactants at the atomic level which provides accurate compositions and constituent phases.
2. Save energy and time.
3. Provide high-purity and well-crystallized final product.
4. Very short reaction time favours the formation of nanocrystalline material. (15-18)

Literature Review

B. PARVATHEESWARA RAO et al. had synthesized Ni-Zn Ferrite by soft chemical approach of co precipitation method. They had observed that with increase in temperature, magnetisation and coercivity increases. (19)

PETRISOR SAMOILA *et al* synthesized nanosized spinel ferrites MFe2O4 (M = Ni, Co, and Zn) which have been prepared by sol-gel autocombustion method using citric acid as a fuel agent. They had observed that In accordance with Langmuir isotherm, the maximum adsorption capacity at 293 K is 14.06mg/g for CoFe2O4 and 17.13 mg/g for NiFe2O4. (20)

N. HARI KUMAR et al synthesized copper substituted nickel zinc ferrite nanoparticle by Citrate-Gel Auto Combustion method at low temperature. There observed results can be explained on the basis of composition and crystal size (21)

MATLI PENCHAL REDDY et al synthesized NiCuZn Ferrites which had been prepared by Microwave Sintering Technique**.** (22)

K.PARVEENA, K. SADHANA had synthesized zinc substituted spinel ferrites using microwave hydrothermal and auto-combustion method.(23)

PEDRO PAULO SILVA ORTEGA et al had synthesized $Y_3Fe_{5-x}Al_xO_{12}$ garnets prepared by the soft chemical method. They had study over the structural, morphological and magnetic properties at room temperature of crystalline aluminium substituted yttrium iron garnet. They had oserved that $Y_3Fe_{5-x}Al_xO_{12}$ (YIG, with $1.5 < x < 1.7$) nanoparticles with no impurities were synthesized by the polymeric precursor method. (24)

YONGMING HU **,**et al had synthesized nanoparticles (NPs) of multiferroic bismuth ferrite ($BiFeO_3$) with narrow size distributions by wet chemical method using bismuth nitrate and iron nitrate as starting materials and excess tartaric acid and citric acid as chelating agent. They had observed that bismuth ferrite $BiFeO_3$ nanoparticles crystallized at 350◦C when using citric acid as chelating agent. Such crystallization temperature is much lower than that of conventional chemical process in which other types of chelating agent are used.$BiFeO_3$ nanoparticles with different sizes distributions show ferromagnetic properties, and the magnetization is increased with reducing the particle size.(25)

MOHAMED. I. M. OMER et al had synthesized $MgFe_2O_4$ magnesium nanoparticle ferrites by wet chemical method using high purity Ferric chloride and Magnesium chloride with oleic acid as the surfactant. They had characterise the structure properties and magnetic properties of $MgFe_2O_4$ by using different spectroscopy technique. (26)

R.R. SINHA et al had synthesized bismuth ferrite nano particles by sol-gel method using citric acid. They had observed that the preparation of bismuth ferrite nano particles does not take place at high temperature as bismuth is lost at high temperature. Thus, temperature conditions are required for preparation of bismuth ferrite nano particles. They characterise the bismuth ferrite nano particle using different techniques. (27)

RAPOLU SRIDHAR et al had synthesized mixed Ni-Cu ferrites by citrate gel technique. (28)

SHIVANI MALHOTRA et al had synthesized strontium ferrite particle by chemical co precipitation method. They had observed that the chemical co-precipitation method for synthesis of strontium hexaferrite nano-material is an efficient and easier technique. The synthesis process has been first carried out without calcination of sample and then with calcinations. They also characterise the properties of strontium ferrite using spectroscopic technique. (29)

M. RAGHASUDHA et al had synthesized magnesium chromium ferrite by citrate gel auto combustion method. They had observed that Citrate Gel auto combustion technique is suitable way for obtaining a homogeneous nano sized mixed Mg-Cr ferrites and the process involves no impurity pickup and material loss. (30)

ILMARS ZALITE et al had synthesized nickel and cobalt ferrite nano powder by sol gel self-combustion method and high frequency plasma chemical synthesis. (31)

.M.M. RASHAD et al had synthesized copper ferrite nanopowder by hydrothermal route using industrial waste**.** (32)

LEENA JASWAL, BRIJESH SINGH had synthesized mixed cobalt zinc ferrite by solid state reaction technique. By this technique they had concluded that the lattice parameter decreases with increasing cobalt content. Magnetic properties show that the prepared sample exhibit ferromagnetic behaviour at room temperature. The saturation magnetization increases with increasing cobalt content. Curie temperature of various samples was calculated. The Curie temperature increases with increasing content of Cobalt ions. (33)

NAWAL KISHORE and S. MUKHERJEE had synthesized mixed ferrite of manganese nickel ferrite by wet chemical co precipitation method. They had observed that the particles were found to be exhibiting a spinel structure with sizes varying from 21nm - 51nm. (34)

RAKESH M. SHEDAM et al had synthesized nano sized cadmium ferrite by oxalate co-precipitation method. They had observed that their characterstics properties including structure and particle size etc. (35)

L. L. WANG and H. Y. He had synthesized cobalt zinc ferrite nano particles by hydrothermal method. They conclude the properties of this type of ferrite using different techniques. (36)
Y.C. GANGADHARAIAH and D. RAVINDER had synthesized cadmium substituted copper nano ferrites by citrate gel auto combustion technique. They had observed that particle size varies for different concentration. Though all the samples were prepared under identical condition, the crystallite size was not the same for all Cd concentrations. This was probably due to the preparation condition followed here which gave rise to different rate of ferrite formation for different concentrations of Cu, favoring the variation of crystallite size. (37)

R. B. BHISE AND S. M. RATHOD had synthesized nickel cobalt zinc ferrite by sol gel auto combustion method. They had observed that Zn and Ni ferrite show more porous nature than

Co ferrite. This is because by varying the concentration of CoFeO and ZnFeO and NiFeO, there is increase in Lattice Constant, inter planer distance, Porosity and decrease in density at increasing temperature. The results of this paper conclude that as porosity increases with respect to increase in temperature and concentration of Co, Ni and Zn ferrites respectively. Thus, the porosity in Zn ferrite is higher than Ni ferrite than Co ferrite. (38)

Y L N MURTHY et al had synthesized nano copper ferrite by citrate gel precursor method. They used the nano copper ferrite as reusable catalyst which is used for synthesis of α, β unsaturated ketone compound. They had observed that nano copper ferrites using as catalyst has advantages are less expensive, heterogeneous reusable catalyst; mild reaction conditions, high yields of products, shorter reaction times, no isomerization during the reaction and easy workup. (39)

ANUCHIT HUNYEK, CHITNARONG SIRISATHITKUL had synthesized cobalt ferrite by sol gel method. He observed that Their magnetic squareness and coercive field were slightly reduced with the increase in the annealing at 800 °C from 2 h to 6 h. To examine the reproducibility of ferrite products of the sol-gel method, the synthetic condition was repeated 28 times. After annealing for 4 h, $CoFe_2O_4$ samples from different batches exhibit variations in the magnetic properties. (40)

MANIK GUPTA et al had synthesized nanosized cesium ferrite by precursor and solution combustion method. In this, pure cesium ferrites were successfully prepared by precursor and solution combustion methods. (41)

J. F. HOCHEPIED and M. P. PILENI had synthesized mixed cobalt zinc ferrite and studied the magnetic properties of cobalt zinc ferrite nano particles. They observed that the magnetization curves show neither hysteresis nor coercivity. This is characteristic of superparamagnetic behaviour. (42)

MANIK GUPTA AND B.S. RANDHAWA had synthesized mixed cesium zinc ferrite by solution combustion method and studied the microstructral, magnetic and electrical properties of mixed ferrite. (43)

LIXIA WANG et al had studied on adsorption of congo red dye on nanocrystalline using spinel ferrite and synthesized spinel ferrite. From the experimental result, Nanocrystalline MFe2O4 ferrites with spinel structure were successfully synthesized by a facile one-step hydrothermal synthesis. (44)

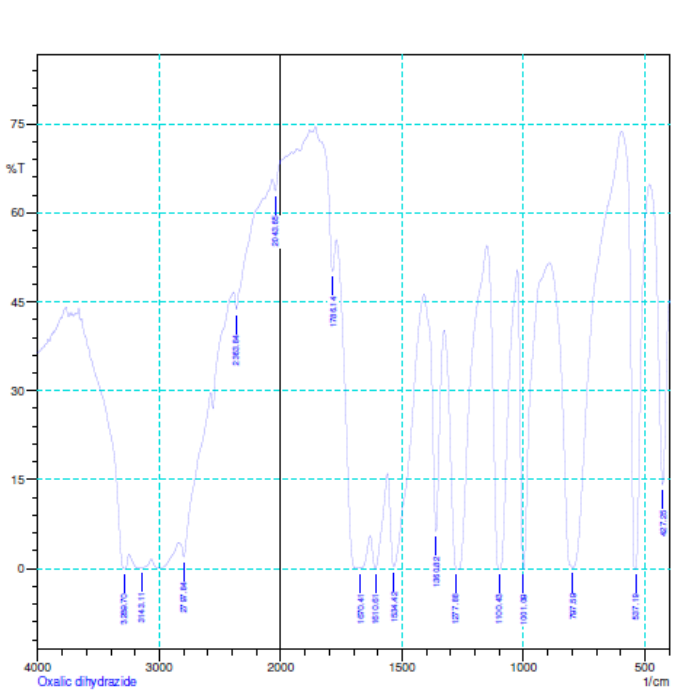
EXPERIMENTAL WORK

PREPARATION METHOD OF OXALYL DIHYDRAZIDE (ODH):

For the preparation of oxalyl dihydrazide, 2 mole of hydrazine hydrate and 1 mole of diethyl oxalate were mixed. Reaction was carried out with dropwise addition of diethyl oxalate to hydrazine hydrate by maintaining the temperature 0-4^0 C, as the reaction is exothermic. White solid precipitates of ODH were formed. The by-product (alcohol) was then evaporated on water bath. Melting point was determine as 240 ^{0}C and compared with literature value. Oxalyl dihydrazide was used as a fuel for combustion synthesis of Cesium ferrite.

Chemical reaction:

$$NH_2.NH_2 .H_2O + (C_2H_5COO)_2 \rightarrow C_2H_5OH + (CONHNH_2)_2$$



No.	Peak	Intensity	Corr. Inte	Base (H)	Base (L)	Area	Corr. Are
1	427.25	14.186	37.795	481.26	399.28	35.874	14.071
2	537.19	0	1.098	538.16	481.26	320.591	-5374.966
3	797.59	0.17	3.878	806.27	653.89	125.704	-60.253
4	1001.09	0	0	1026.16	1001.09	498.923	-2012.353
5	1100.43	0	0	1102.35	1100.43	385.776	0
6	1277.88	0	0	1326.1	1277.88	331.335	-4500.404
7	1360.82	6.34	36.39	1410.01	1326.1	49.056	18.427
8	1534.42	0.395	20.876	1560.46	1410.01	142.699	57.736
9	1610.61	0	8.658	1631.83	1560.46	299.726	226.514
10	1670.41	0.085	0.059	1680.05	1668.48	32.959	0.784
11	1786.14	50.185	9.282	1831.47	1771.68	12.86	0.946
12	2043.65	63.727	3.17	2068.72	2003.14	11.896	0.601
13	2363.84	43.735	4.462	2387.95	2141.06	66.012	-0.108
14	2797.84	2.004	6.57	2841.24	2576.02	250.172	0.909
15	3143.11	0.242	0.008	3147.93	3140.22	20.112	0.048
16	3289.7	0.067	0.123	3581.93	3288.74	235.432	-291.461

IR SPECTRUM OF OXALYL DIHYDRAZIDE (EXPERIMENTAL ANALYSIS)

1) 2^{0}amine observed at approximately 3286cm^{-1}(stretching)

2) 1^0 amine=3143cm^{-1}(stretching).

3) Amide group=1670cm^{-1}(CO-NH)(due to conjugation ,value decreases).

4) Primary amine =1670cm^{-1}(bending).

5) Secondary amine=1610cm^{-1}(bending).

6) C-N Stretching=1360cm^{-1}.

7) C-C Stretching=1277$cm^{-1.}$

8) C-O Stretching=1100cm^{-1}(due to conjugation).

SYNTHESIS OF CESIUM FERRITE:

Cesium ferrite having the molecular formula $CsFeO_2$ was prepared by using solution phase combustion method. In the combustion method, Solution combustion is an exciting phenomenon, which involves propagation of self-sustained exothermic reactions. This process allows for the synthesis of a variety of nanoscale materials, including oxides, metals, alloys, and sulphides.

1) 1 mole of cesium nitrate in 500ml beaker was dissolved in minimum quantity of distilled water (Solution A).

2) 1 mole of ferric nitrate in 500 ml beaker was dissolved in minimum quantity of distilled water (Solution B).

3) Solution A and B were mixed together.

4) ODH (2 moles) was added in above solution very slowly and with proper mixing as the reaction is very exothermic.

5) After complete addition of ODH, viscous solution then obtained was concentrated on water bath.

6) The concentrate was then subjected to heating in a muffle furnace by gradually increasing the temperature (step by step) up to 600^0C. Brown coloured product was obtained and this was grinded further by using mortar and pestle to obtain fine powder.

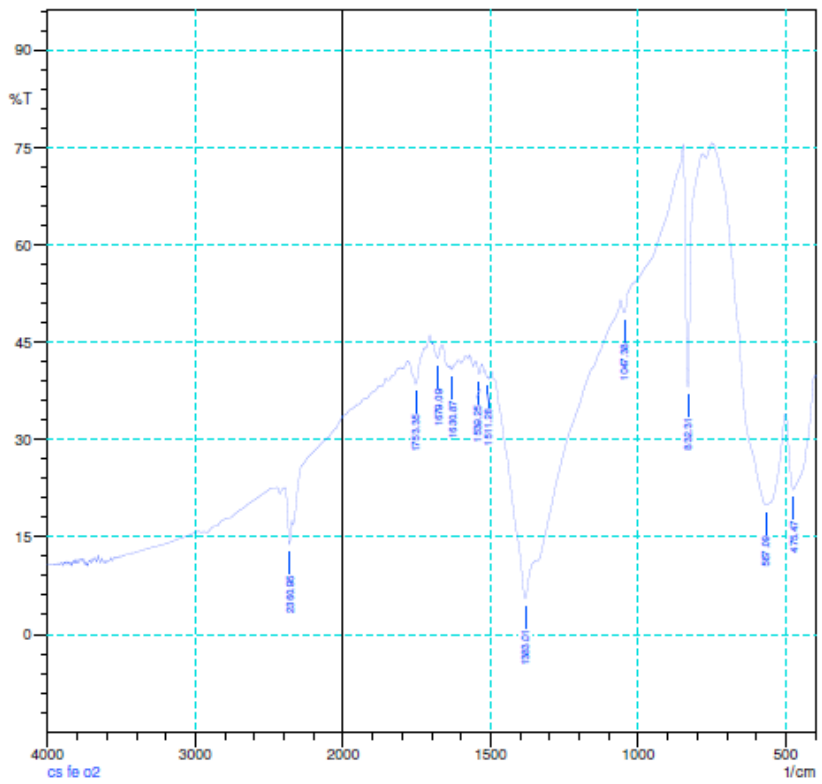
Reaction involves:

$Fe(NO_3)_3.9H_2O + CsNO_3 + 2C_2H_6N_4O_2 \rightarrow CsFeO_2 + 4CO_2\,(g) + 6N_2\,(g) + 15H_2O$

THEORETICAL YIELD: 7.36

ACTUAL YIELD: 7.31

PERCENTAGE YIELD: 99.3%



No.	Peak	Intensity	Corr. Inte	Base (H)	Base (L)	Area	Corr. Are
1	475.47	22.304	13.262	501.51	406.03	53.82	12.32
2	567.09	19.954	22.762	680.89	501.51	97.695	34.346
3	832.31	38.072	37.028	847.74	783.13	13.489	5.319
4	1047.38	49.576	3.446	1060.88	847.74	48.051	4.29
5	1383.01	5.527	12.766	1485.24	1349.25	105.917	14.122
6	1511.28	39.534	0.436	1518.99	1508.38	4.234	0.034
7	1539.25	40.082	1.657	1546.96	1531.53	6.018	0.162
8	1630.87	40.808	0.561	1634.73	1622.19	4.832	0.033
9	1679.09	42.58	1.792	1696.45	1671.37	9.077	0.26
10	1753.35	38.579	4.551	1779.39	1726.35	20.559	1.19
11	2360.95	13.831	5.334	2393.74	2345.52	36.301	2.512

IR SPECTRUM OF $CsFeO_2$:

PREPARATION OF MIXED CESIUM LITHIUM FERRITE:

Mixed Cesium-Lithium ferrite ($Cs_{0.5}Li_{0.5}FeO_2$) was synthesized by solution combustion method following the same steps.

Reaction:

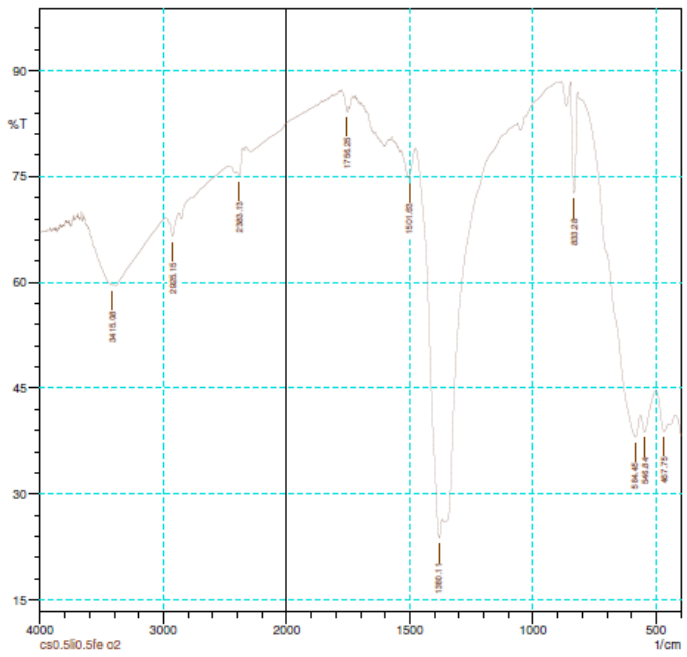
$LiNO_3 + Fe(NO_3)_3.9H_2O + 2C_2H_6N_4O_2 + CsNO_3 \rightarrow Cs0.5Li0.5FeO2 + 4CO_2\,(g) + 6N_2\,(g)$

$+15H_2O.$

THEORETICAL YIELD: 7.87

ACTUAL YIELD: 7.83

PERCENTAGE YIELD: 99.4%



No.	Peak	Intensity	Corr. Inte	Base (H)	Base (L)	Area	Corr. Are
1	467.75	38.854	2.817	502.47	447.5	21.324	0.738
2	546.84	38.804	3.336	564.2	502.47	23.643	0.932
3	584.45	38.077	6.905	801.45	564.2	55.972	2.386
4	833.28	72.656	15.005	848.71	819.77	2.973	1.325
5	1380.11	23.773	7.986	1473.66	1369.5	33.726	-1.776
6	1501.63	74.831	0.382	1503.56	1477.52	2.961	-0.006
7	1756.25	84.218	0.33	1776.5	1754.32	1.464	-0.015
8	2383.13	75.126	2.345	2413.03	2357.09	6.626	0.383
9	2925.15	66.54	2.917	2980.12	2871.14	17.997	0.744
10	3415.08	59.592	0.023	3417.98	3407.37	2.384	0.001

IR SPECTRUM OF $Cs_{0.5}Li_{0.5}FeO_2$

RESULT AND DISCUSSION AND FUTURE PLAN

Identity of ODH and ferrite materials have been established by IR spectroscopy. Purity of synthesized ferrite materials will be checked by X-ray powder diffraction. For morphology and particle size analysis SEM/TEM studies will be carried out. These ferrite materials will be further used for adsorption of various dyes from the solution and effect of various parameters on adsorption e.g. variation of concentration, pH etc. will be reported.

CONCLUSION

Oxalyl dihydrazide acts as a fuel for combustion synthesis. This method has many advantages over conventional methods. Ferrite materials having single phase can be synthesized as a result of stoichiometric mixing of metal nitrates and oxalyl dihydrazide. Ferrite materials are synthesized in shorter time. Nano sized ferrite materials can also be synthesized as this method involves atomic scale mixing of reacting species.

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