

**Completion Report of Minor Research Project**

MRP Sanction letter No: MRP(S)-0425/13-14/KAGU009/UGC-SWRO dated 08-Apr-2014

Project Title

***SYNTHESIS AND CHARECTERIZATION OF LITHIUM-  
ZINC FERRITES***

Submitted by

**Dr. Vinodkumar Rathod**

**Principal Investigator**



**GOVERNMENT COLLEGE (AUTONOMOUS), KALABURAGI-585105,  
KARNATAKA, INDIA**

**From:**

**Dr. VINODKUMAR RATHOD**

Principal Investigator,  
Assistant Professor of Physics,  
Government College (Autonomous),  
Kalaburagi (Karnataka).

**Date: 30-11-2019**

**To**

**The Deputy Secretary,  
SWRO-UGC,  
Bangalore.**

**Subject:** Submission of Final Report of UGC Minor Research Project in physics  
Ref:- File No: MRP(S)-0425/13-14/KAGU009/UGC-SWRO, Dated 28-Mar-2014

Sir,

I am here by submitting the final report of my UGC minor research project in Physics, along with utilization certificate (File No: MRP(S)-0425/13-14/KAGU009/UGC-SWRO, Dated 08-Apr-2014) entitled "SYNTHESIS AND CHARECTERIZTION OF LITHIUM-ZINC FERRITES." This is for your kind information and necessary action. Hence, I request you to kindly release the remaining financial grant sanctioned towards the said project at the earliest.

Thanking You,

Yours Sincerely



**Dr. Vinodkumar Rathod**  
Principal Investigator &  
Assistant Professor in Physics.  
Govt. College (Autonomous) Kalaburagi

Government of Karnataka

(Department of Collegiate Education)

EST/GCK/UGC-MRP/2013-14/ 361

Office of the Principal  
Govt. College (Autonomous) Kalaburagi  
Date: 30-11-2019

The final report on the UGC Minor Research Project of Dr. Vinodkumar Rathod is forwarded for your needful action.



**PRINCIPAL**

Govt. College (Autonomous)  
KALABURAGI-585102

## Completion Report of Minor Research Project

MRP Sanction letter No: MRP(S)-0425/13-14/KAGU009/UGC-SWRO dated 08-Apr-2014

Project Title

**SYNTHESIS AND CHARECTERIZATION OF LITHIUM ZINC  
FERRITES**

Submitted by

**Dr. Vinodkumar Rathod**

**Principal Investigator**



**GOVERNMENT COLLEGE (AUTONOMOUS), KALABURAGI-585105,  
KARNATAKA, INDIA**

**Date: 30-11-2019**

  
**Signature of the  
Principal Investigator**

**Dr. Vinodkumar Rathod**  
M.Sc. M.Phil. Ph.D.  
Asst. Professor of Physics  
Govt. College (Autonomous) KALABURAGI-05

  
**Principal  
PRINCIPAL**  
Govt. College (Autonomous)  
KALABURAGI-585102

To,  
The Account Officer,  
SWRO University Grants Commission,  
P.K. Block, Palace road, Gandhi Nagar,  
Bengaluru-560009.

Date:30-11-2019

(Through the Principal Govt. College, Kalaburagi-585105)

Sub: Submission of Accounts of Expenditure for the Minor Research Project MRP  
Sanction letter No. MRP(S)-0425/13-14/KAGU009/UGC-SWRO dated 08-Apr-2014

With Reference to the above cited subject, I am herewith submitting the detail of expenditure incurred for completing the Minor Research Project (MRP) sanctioned by UGC. The details are as follows

SlNo.	Items	UGC Sanctioned amount Rupees	Grant received Rupees	Expenditure incurred Rupees
1	<b>Non-Recurring:</b> Books and Journals	10,000=00	10000=00	10,000=00
2	<b>Recurring:</b> Contingency including special needs	20,000=00	10,000=00	20,000=00
	Chemicals and Glasswares	40,000=00	20,000=00	40,000=00
	Field and travel	5,000=00	2,500=00	5,000=00
	<b>Total</b>	<b>75,000=00</b>	<b>42,500=00</b>	<b>75,000=00</b>

The UGC Sanctioned amount Rs 75,000=00  
Total expenditure incurred for completion of MRP Rs 75,000=00  
Grant received from UGC Rs 42,500=00  
**The balance Grant to be released Rs 32,500=00**

Hence I request you to release the balance of Rs 32,500=00 at an earliest possible.

Yours faithfully

  
(Dr. Vinodkumar Rathod)

- Encl:** 1. Covering letter to Deputy Secretary, SWRO, Bangalore  
2. Utilization certificate in Annexure-V  
3. Annexure III, VI and VII  
4. UGC MRP sanction letter

**Dr. Vinodkumar Rathod**  
M.Sc. M.Phil. Ph.D.  
Asst. Professor of Physics  
Govt. College (Autonomous) KALABURAGI-05

  
**PRINCIPAL**  
Govt. College (Autonomous)  
KALABURAGI-585102

## Annexure – VI

**UNIVERSITY GRANTS COMMISSION  
BAHADUR SHAH ZAFAR MARG  
NEW DELHI – 110 002**

**ANNUAL/FINAL REPORT OF THE WORK DONE ON THE MINOR RESEARCH PROJECT  
(Report to be submitted within 6 weeks after completion of each year)**

1	Project Report No. 1 <sup>st</sup> /2 <sup>nd</sup> /3 <sup>rd</sup> /Final	Final
2	UGC Reference No.	No. MRP(S)-0425/13-14/KAGU009/UGC-SWRO dated 08-Apr-2014
3	Period of report	Two years From 28-03-2014 to 28-03-2016
4	Title of the research project	Synthesis and characterization of Lithium-Zinc ferrites
5	a. Name of the Principal investigator	Dr. Vinodkumar Rathod
	b. Dept. and university/college where work has progressed	Dept. of Physics, Govt. College, Kalaburagi-585105.
6	Effective date of starting the project	28 March 2014
7	Grant Approved and expenditure incurred during the period of the report:	
	Total amount approved Rs	Rs. 75,000=00
	Total Grant received Rs.	Rs. 42,500=00
	FINAL EXPENDITURE	Rs.75,000=00
8	Report of the work done: (Please attach a separate sheet)	Enclosed
i.	Brief objectives of the project	Enclosed Annexure VIII-1
ii.	Work done so far and results achieved and publications, if any resulting from the work( Give details of the papers and names of the journals in which it has been published or accepted for publication)	Yes, Details given in Annexure VIII-2
iii.	Has the progress been according to original plan of work and towards achieving the objective, if not state reasons	Yes.
iv.	Please indicate the difficulties, if any experienced in implementing the project	No
v.	If Project has not been completed, please indicate the approximate time by which it is likely to be completed. A summary of the work done for the period (Annual Basis) may please be sent to the commission on a separate sheet	Completed

vi.	If the project has been completed, please enclose a summary of the findings of the study. Two bound copies of the final report of work done may also be sent to the Commission.	Enclosed
vii.	Any other information which would help in evaluation of work done on the project. At the completion of the project, the first report should indicate the output, such as a) Manpower trained (b) Ph.D Awarded (c) Publication of results (d) other impact if any	No

  
(PRINCIPAL INVESTIGATOR)

**Dr. Vinodkumar Rathod**  
M.Sc. M.Phil. Ph.D.  
Asst. Professor of Physics  
Govt. College (Autonomous) KALABURAGI-05

  
(PRINCIPAL)

**PRINCIPAL**  
Govt. College (Autonomous)  
KALABURAGI-585102



# AVANTI PATIL & ASSOCIATES

**CHARTERED ACCOUNTANTS**

# 3-30/3 - Laxminarayana Complex - Saraswati Godam - Super Market  
Main Road, KALABURAGI - 585 101 - E-mail : casgavanti@yahoo.in  
Tel. : 08472-224733 - Mob. : 9482549103

Date 29/11/2019

Ref No.

**Annexure - V**

## UTILIZATION CERTIFICATE

Certified that the grant of Rs. 75,000=00 (Rupees SEVENTY FIVE THOUSAND only) received from the University Grants Commission under the scheme of support for Minor Research Project entitled "SYNTHESIS AND CHARECERIZATION OF LITHIUM-ZINC FERRITES" vide UGC letterNo.MRP(S)-0425/13-14/KAGU009/UGC-SWRO dated 28/03/2014 has been fully utilized for the purpose for which it was sanctioned and in accordance with the terms and conditions laid down by the University Grants Commission.

If as a result of check or audit objection, some irregularity is noticed at a later stage, action will be taken to refund or regularize the objected amount. It is further certified that inventories of permanent or semi-permanent assets created/acquired wholly or mainly out of the grants given by the University Grants Commission as indicated above are being maintained in the prescribed form and are being kept up to date and these assets have not been disposed of encumbered or utilized for any other purpose.

Place: Kalaburagi

SIGNATURE OF THE  
PRINCIPAL INVESTIGATOR

**Dr. Vinodkumar Rathod**  
M Sc. M.Phil. Ph.D.

Asst. Professor of Physics  
Govt. College (Autonomous) KALABURAGI-05



PRINCIPAL

Govt. College (Autonomous)  
KALABURAGI-585102

**For Avanti Patil & Associates  
Chartered Accountants**

(Shambhulingappa G. Avanti)  
Partner

STATUTORY AUDITOR

(Seal)

UDIN: 19224611AAAAEK8990

**GOVERNMENT DEGREE COLLEGE (MRP)(S)**

**SEDEM ROAD KALABURGI-585105**

**RECCEIPTS ANDPAYMENTSACCOUNT FOR THE YEAR END**

RECEIPTS	AMOUNT	PAYMENTS	AMOUNT
<b><u>To Opening Balance</u></b>		<b><u>By Non-Recurring</u></b>	
Cash on Hand	--	Books & Journals	10,000.00
Cash on bank	--		
<b><u>Grant Received from</u></b>		<b><u>Recurring</u></b>	
MRP(S)-0425/13-	42,500.00	Contingency	20,000.00
14/KAGU009/UGC/SWRO Dated		Chemicals & glassware	20,000.00
28/03/2014		Field work & Travel	5,000.00
Hand loan	32,500.00		--
		Closing Balance	--
		Cash on hand	--
		Cash at Bank	--
<b>Total</b>	<b>75,000.00</b>	<b>TOTAL</b>	<b>75,000.00</b>

Date: 29/11/2019

Place: Kalaburagi



For Avanti Patil & Associates  
Chartered Accountants

(Shambhulingappa G. Avanti)  
Partner

*[Signature]*  
PRINCIPAL  
Govt. College (Autonomous)  
KALABURAGI-585102

**Annexure - III**

**UNIVERSITY GRANTS COMMISSION  
BAHADUR SHAH ZAFAR MARG  
NEW DELHI - 110 002**

**STATEMENT OF EXPENDITURE IN RESPECT OF MINOR RESEARCH PROJECT**

1. Name of Principal Investigator  
2. Dept. of Principal Investigator University/College  
3. UGC approval Letter No. and Date  
4. Title of the Research Project  
5. Effective date of starting the project  
6. a. Period of Expenditure  
b. Details of Expenditure

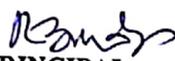
Dr. Vinodkumar Rathod  
Dept. of Physics,  
Govt. College, Kalaburagir-585105  
MRP(S)/13-14/KAGU009/UGC-SWRO  
dated 15- Feb-2014  
**Synthesis and Characterization of Lithium-Zinc Ferrites**  
28 March 2014  
From 28 March 2014 to 28 Nov 2019

Sl.No.	Items	UGC Sanctioned amount Rupees	Grant received Rupees	Expenditure incurred Rupees
1	<b>Non-Recurring:</b> Books and Journals	10,000=00	10000=00	10,000=00
2	<b>Recurring:</b> Contingency including special needs	20,000=00	10,000=00	20,000=00
	Chemicals and Glasswares	40,000=00	20,000=00	40,000=00
	Field and travel	5,000=00	2,500=00	5,000=00
	<b>Total</b>	<b>75,000=00</b>	<b>42,500=00</b>	<b>75,000=00</b>

c. Staff Date of Appointment: Not Applicable.

It is certified that the grant of Rs. 42,500/- (Rupees Fourty two thousand and five hundred only) received from the sanctioned amount Rs 75,000/ - (Seventy five thousand only) from the University Grants Commission under the scheme of support for Minor Research Project entitled **Synthesis and Characterization of Lithium-Zinc Ferrites** vide UGC letter No. MRP(S)-0425/13-14/KAGU009/UGC-SWRO dated 28- Mar-2014 have been fully utilized for the purpose for which it was sanctioned and in accordance with the terms and conditions laid down by the University Grants Commission.

  
**PRINCIPAL INVESTIGATOR**  
Dr. Vinodkumar Rathod  
M.Sc. M.Phil. Ph.D.  
Asst. Professor of Physics  
Govt. College (Autonomous) KALABURAGI-05

  
**PRINCIPAL**  
PRINCIPAL  
Govt. College (Autonomous)  
KALABURAGI-585102

Annexure - IV

UNIVERSITY GRANTS COMMISSION  
BAHADUR SHAH ZAFAR MARG  
NEW DELHI - 110002

STATEMENT OF EXPENDITURE INCURRED ON FIELD WORK

Name of the Principal Investigator: Dr. Vinodkumar Rathod

Name of the Place visited	Duration of the Visit		Mode of Journey	Expenditure Incurred (Rs.)
	From	To		
IISC Bangalore	09-06-2014	12-06-2014	Train	395
IISC Bangalore	27-04-2015	29-04-2015	Train	1002
IISC Bangalore	08-02-2016	11-02-2016	Train	730
IISC Bangalore	16-11-2016	19-11-2016	Train	722
IISC Bangalore	11-12-2016	13-12-2016	Train	1492
IISC Bangalore	23-05-2017	25-05-2017	Bus	1040
TOTAL				5381

Certified that the above expenditure is in accordance with the UGC norms for Minor Research Projects.



PRINCIPAL INVESTIGATOR

**Dr. Vinodkumar Rathod**  
M.Sc. M.Phil. Ph.D.  
Asst. Professor of Physics  
Govt. College (Autonomous) KALABURAGI-05

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KALABURAGI-585102

Annexure – VII

UNIVERSITY GRANTS COMMISSION  
BAHADUR SHAH ZAFAR MARG  
NEW DELHI – 110 002

PROFORMA FOR SUBMISSION OF INFORMATION AT THE TIME OF SENDING THE  
FINAL REPORT OF THE WORK DONE ON THE PROJECT

1	NAME AND ADDRESS OF THE PRINCIPAL INVESTIGATOR	Dr. Vinodkumar Rathod, Assistant professor of Physics, Govt college Kalaburagi
2	NAME AND ADDRESS OF THE INSTITUTION	Department Of Physics, Govt.college, Kalaburagi-585105 Karnataka
3	UGC APPROVAL NO. AND DATE	No. MRP(S)-0425/13-14/KAGU009/UGC-SWRO dated 28- Marr-2014
4	DATE OF IMPLEMENTATION	28 March 2014
5	TENURE OF THE PROJECT	Two years From 28-03-2014 to 28-03-2016
6	TOTAL GRANT ALLOCATED	Rs. 75,000=00
7	TOTAL GRANT RECEIVED	Rs. 42,500=00
8	FINAL EXPENDITURE	Rs.75,000=00
9	TITLE OF THE PROJECT	<b>Synthesis and Characterization of Lithium-Zinc Ferrites</b>
10	OBJECTIVES OF THE PROJECT	Enclosed Annexure –VIII-1
11	WHETHER OBJECTIVES WERE ACHIEVED (GIVE DETAILS)	Yes (Details given in Annexure VIII-2)
12	ACHIEVEMENTS FROM THE PROJECT	Required Chemicals were brought. The samples were synthesized and XRD, SEM, EDAX, FTIR characterizations were carried out.
13	SUMMARY OF THE FINDINGS ( IN 500 WORDS )	Enclosed Annexure-VIII-3
14	CONTRIBUTION TO THE SOCIETY ( GIVE DETAILS )	
15	WHETHER ANY PH.D.ENROLLED/PRODUCED OUT OF THE PROJECT	YES
16	NO. OF PUBLICATIONS OUT OF THE PROJECT	02

  
(PRINCIPAL INVESTIGATOR)

**Dr. Vinodkumar Rathod**  
M.Sc. M.Phil. Ph.D.  
Asst. Professor of Physics  
Govt. College (Autonomous) KALABURAGI-05

  
(PRINCIPAL)  
PRINCIPAL  
Govt. College (Autonomous)  
KALABURAGI-585102



Govt. of Karnataka  
Department of Collegiate Education  
**GOVERNMENT COLLEGE (AUTONOMOUS)**  
**KALABURAGI-585105**



Ph: 08472 - 245064. Website: <http://gc.kar.nic.in/kalaburagi/>  
Email : [principal.gc.kalaaburagi@gmail.com](mailto:principal.gc.kalaaburagi@gmail.com)

Date: 25-11-2019

**CERTIFICATE**

1. It is certified that, a copy of Final report of Minor research Project (MRP) entitled "SYNTHESIS AND CHARECTERIZATION OF LITHIUM-ZINC FERRITES" submitted by Dr. Vinodkumar Rathod , Assistant Professor , Dept. of Physics, has been kept in the college Library.
2. An executive summary of the Minor research Project (MRP) entitled "SYNTHESIS AND CHARECTERIZATION OF LITHIUM ZINC FERRITES" has been uploaded in the college website: <http://gc.kar.nic.in/kalaburagi/>

  
Principal  
**PRINCIPAL**  
Govt. College (Autonomous)  
KALABURAGI-585102

**Introduction of the Project:**

Pure and substituted lithium ferrites are low cost materials used for a wide variety of technological applications [1-6]. Lithium ferrite finds applications in many high frequency electronic devices such as in microwave circulators, isolators, phase shifters and absorbers, because of its high permeability (in microwave frequency range) and high Curie temperature. The other properties which make Li-ferrite suitable for such applications are its high electrical resistivity, mechanical strength and chemical stability [2, 7]. Lithium ferrite is often doped with other cations to optimize its electrical, magnetic, and elastic properties [2]. Previously, non-magnetic ion substituted lithium ferrites were prepared using different methods such as conventional double sintering [8], sol-gel [9-11] and solid-state reaction of inorganic precursors [12] etc. Synthesis of lithium ferrites using these methods is either, time and power consuming, or cumbersome. In addition, in solid state reaction route, high calcination temperature lowers the magnetization due to precipitation of  $\alpha$ - $\text{Fe}_2\text{O}_3$ , or formation of  $\text{Fe}_3\text{O}_4$  phase along with a non-magnetic phase (e.g., ZnO in case of Li-Zn ferrites). Furthermore, the reduction of  $\text{Fe}^{3+}$  to  $\text{Fe}^{2+}$  (during the formation of  $\text{Fe}_3\text{O}_4$ ) can lead to an increased electrical conductivity limiting these materials in microwave applications, where high resistivity and minimum dielectric loss are required [7].

Previously, Li-Zn ferrite powders were prepared by simple methods such as microwave-induced glycine-nitrate process [13, 14] and solution combustion [15]. These methods require nitrate precursors which are hygroscopic, not always commercially available and often expensive than oxide precursors. To overcome these limitations, we have used 'combustion synthesis' method [16-17], which is an advanced approach in powder metallurgy. In this method, the oxide precursors (solids) and the fuel are homogeneously mixed followed by a rapid exothermic

reaction. The reaction is initiated by heating the precursor materials to a lower temperature, e.g., ~ 250 °C. The fuel is then ignited at its flash point and the reaction self-sustains due to its exothermic nature. Here, for the first time we have used combustion synthesis method to synthesize Li-Zn ferrite powders using polyethylene glycol (PEG) as fuel. As PEG contains saturated bonds, it provides more energy during its combustion and facilitates the formation of, otherwise difficult to form, Li-Zn ferrite. In this technique, like a few of the above techniques (e.g., solution combustion), nanoscale powders of desired composition can be synthesized in one step, avoiding additional calcination, because the heat generated due to burning of the fuel is sufficient to form the desired phase [7]. Hence, the combustion synthesis method is promising for the synthesis of wide varieties of powders with fine microstructures and tailored properties.

Lithium ferrites are spinel ferrites having general formula  $AB_2O_4$ . For spinels this formula is generally represented as  $(A)[B_2]O_4$ , where the parenthesis represents the tetrahedral site and the square bracket represents the octahedral site. In normal spinel ferrites the tetrahedral (A-) site is occupied by a divalent cation (such as  $Zn^{2+}$ ,  $Mg^{2+}$  etc.) and octahedral (B-) site is occupied by trivalent cation (such as  $Fe^{3+}$ ). In inverse spinel ferrites, the divalent cations (such as  $Ni^{2+}$ ) occupy the B site. In some ferrites, the occupation of divalent cations is distributed in A- and B-sites: these ferrites are known as mixed spinel ferrites. Our studied samples, Zn-doped lithium-ferrites, are typical examples of mixed spinel ferrites of the type  $(Zn_xFe_{1-x})[\{Li_{(1-x)/2}Fe_{(1-x)/2}\}Fe_{(1+x)}]O_4$ , where the  $Zn^{2+}$  cation goes to the A-site and the combination of cations,  $[Li^{+}_{0.5}Fe^{3+}_{0.5}]$ , which forms the divalent-metal cation, goes to the octahedral (B-) sites. The rest of the  $Fe^{3+}$  cations are distributed in the A- and B-sites. The distribution of different cations at the interstitial sites modifies the infrared absorption spectrum, as the dipole moment change during vibration of the cation complexes varies due to different mass, charge and strength of interaction between A- and B-site complexes.

The mechanical properties of materials can be understood from the nature of the chemical bond and their vibrational properties. In general, most of the properties such as mechanical, vibrational, elastic and thermal etc. depend on the nature of binding forces between the atoms in the materials. As the binding forces depend on the type of atoms/ions and the inter-atomic/inter-ionic forces involved, the substitution of host element by dopants invariably result in varied physical properties. Hence, lithium ferrite is often doped with other cations to tune its electrical, magnetic, and elastic properties [18].

Infrared (IR) spectroscopy deals with the interaction between a molecule and IR radiation, which causes the excitation of the vibrations (stretching and bending) of bonded atoms within the molecule. The vibrations of cations which are bonded to the oxygen ions in ferrites produce different vibrational frequencies that appear as IR absorption bands in the FTIR spectrum. Hence, the position of the spectral bands depends on the type and strength of bonding, coordination between the ions involved, crystal/local symmetry and type (mass) of the ions involved. The principle of FTIR spectroscopy can be well understood using spring-model of vibration of atoms, wherein the wave number  $\nu$  is defined as:

$$\nu = \frac{1}{2\pi c} \sqrt{k/\mu} \quad (1)$$

where  $k$  is the spring (force) constant,  $\mu$  is the reduced mass of ions involved and  $c$  is the velocity of light. The force constant is a measure of bond strength. The mechanical (elastic), thermal and vibrational properties are closely related to each other and these properties can be determined from the frequencies of vibrations of the atoms and the force constants between the vibrating atoms. As the force constant is an important quantity controlling elastic properties of materials, it is quite interesting to obtain the force constants ( $k$ ) and other elastic moduli of Li-Zn ferrites, keeping in mind their potential mechanical, thermal and industrial applications [19-22].

The elastic and thermal properties of Li-Zn ferrite ( $\text{Li}_{0.5-x/2}\text{Zn}_x\text{Fe}_{2.5-x/2}\text{O}_4$ ) powder samples have been determined from FTIR spectrum. Using the reduced mass ( $\mu$ ) and observed absorption frequencies (in the FTIR spectrum), the force constant ( $k$ ) was calculated from the spring model of atomic vibrations (eq. 1). Other mechanical parameters such as elastic wave velocity, elastic moduli and Debye temperature ( $\theta_D$ ) were also calculated based on these results. The Debye temperature ( $\theta_D$ ) is an important quantity in understanding the vibrational and

thermal properties of the materials which depends on the vibrational density of states and the vibrational amplitude of the oscillating atoms. However, we have calculated the Debye temperature of Li-Zn ferrite samples using two different formalisms based on the FTIR spectral results as will be discussed in the following sections. The results, especially the Debye temperature, were compared with the experimental results available on similar materials [23] obtained using the ultrasonic pulse transmission (UPT) technique.

### **Objectives**

- To synthesize and characterize Lithium Zinc Ferrites,
- To study the structure, Vibrational Spectroscopy, Elastic-thermal and magnetic properties, of Lithium Zinc Ferrites.

### **Methodology**

- To synthesize the Lithium Zinc ferrites by the Combustion Synthesis technique,
- To characterize the Lithium Zinc ferrites by employing XRD, SEM, TG-DSC, FTIR, PFM and Mössbauer spectroscopy.
- To determine density of Lithium Zinc ferrites.
- Rietveld refinements of the structural parameters were carried out using the program "*FullProf*"

## Annexure VIII-2

### Summary of the findings (Brief Report):

Lithium zinc ferrites with the general formula  $\text{Li}_{0.5-x/2}\text{Zn}_x\text{Fe}_{2.5-x/2}\text{O}_4$  ( $x = 0.0, 0.2, 0.4, 0.6, 0.8$  and  $1.0$ ) were prepared using combustion synthesis method. Stoichiometric amount of precursor materials  $\text{Li}_2\text{CO}_3$ ,  $\text{ZnO}$  and  $\text{Fe}_2\text{O}_3$  (all of AR grade) were mixed and thoroughly ground in agate mortar and pestle for  $\sim 1$  h. PEG (MW = 6000) was added to the precursor mixture with a precursor to PEG weight ratio of about 1:2 and the grinding process was further continued for about 2 h. The resulting mixture was taken in an alumina crucible and heated to about  $450^\circ\text{C}$  inside the furnace. Initially the PEG melted at around  $75 - 80^\circ\text{C}$ , the exothermic reaction started (at  $\sim 300 - 450^\circ\text{C}$ ), progressed and completed in about a few minutes, which produced the required Li-Zn ferrite powder. The powders were retained inside the furnace at that temperature ( $\sim 450^\circ\text{C}$ ) for 4 h. The resulting powder was then used for characterizations.

Different stages involved during progress of combustion synthesis of Li-Zn ferrite samples were monitored by thermo-gravimetric analysis (TGA) and differential scanning calorimetry (DSC). The detailed procedure used for the synthesis of the samples is described [24].

The scanning electron micrographs of the synthesized samples show foamy network type of morphology with rod-shaped particles. Rietveld refinement of the X-ray diffraction (XRD) data confirmed the preferential occupancy of the Zn-atoms at the tetrahedral sites and the Li atoms at the octahedral sites of the spinel structure. Trace amounts of zinc-oxide and iron-oxide impurity phases were found for the samples with high Zn content ( $x \geq 0.6$ ). The comparison of the particle sizes obtained from scanning electron micrographs and the crystallite sizes estimated from XRD results suggest that the particles are polycrystalline aggregates of a few crystalline grains. Fourier transform infrared (FTIR) spectroscopy confirmed the presence of broad (vibrational) absorption bands between  $\sim 300\text{-}500\text{ cm}^{-1}$  ( $\nu_1$ ) corresponding to tetrahedral complexes and between  $\sim 530\text{-}800\text{ cm}^{-1}$  ( $\nu_2$ ) corresponding to octahedral complexes in the spinel structure of the Li-Zn ferrites. The phase compositions of the samples were obtained from the analysis of Mössbauer spectra and XRD patterns. Substitution of higher amount of  $\text{Zn}^{2+}$  ions reduces the saturation magnetization of the samples. Our work demonstrates that combustion synthesis method is a cost effective and simple way to synthesize bulk amount of Li-Zn ferrite samples

with desired physical properties. The details about the obtained structural parameters and the content of the phases is described [24].

The FTIR spectrum for each of the sample consists of two broad absorption bands: one at  $\sim 510 - 800 \text{ cm}^{-1}$  corresponding to the vibrations of tetrahedral complexes ( $\nu_1$ ) and the other, at  $\sim 250 - 510 \text{ cm}^{-1}$  corresponding to the vibrations of octahedral-complexes ( $\nu_2$ ) in the spinel structure of the Li-Zn ferrites. Each of these band is further split into four sub-bands. Based on the structure and the occupancy of the cations we have explained the observed infrared absorption bands. Pronounced splitting of the  $\nu_1$  and  $\nu_2$  bands in pure  $\text{Li}_{0.5}\text{Fe}_{2.5}\text{O}_4$  were observed due to the  $\text{LiO}_6$  complexes partially replacing the  $\text{FeO}_6$  complexes at the octahedral interstitial sites. The vibrational frequencies of the  $\text{LiO}_6$  complexes are different from  $\text{FeO}_6$  complexes due to the difference in their low reduced mass and valence of the cations (lower bond strength). The resolved bands smear out with increasing Zn content ( $x$ ) owing to higher statistical disorder in the occupancy of the cations at the tetrahedral and octahedral sites. We observe that the vibrations of the tetrahedral and octahedral complexes are not isolated but rather correlated. Our work helps in understanding the correlation of cation occupancy with the vibrational properties of nanocrystalline Li-Zn ferrites. The experimentation is discussed in detail. The vibrational modes corresponding to the sub bands has been discussed in detail [25].

Composition dependent elastic and thermal properties were derived from the analysis of the Fourier transform infrared (FTIR) spectra measured at RT. Each FTIR spectrum consists of two broad absorption bands corresponding to the vibrations of octahedral- and tetrahedral-complexes in the spinel structure of the Li-Zn ferrites. We observed pronounced splitting of both the bands of pure  $\text{Li}_{0.5}\text{Fe}_{2.5}\text{O}_4$  due to the presence of light weight  $\text{Li}^+$  ions at the octahedral sites. However, the splitting smears out with increasing Zn content ( $x$ ) due to statistical disorder in the occupancy of the cations. Using the IR absorption frequencies, the lattice parameters and the mass-densities of our samples, we evaluated the elastic and thermal parameters of the studied Li-Zn ferrite samples as a function of Zn-content and compared those with available experimental results. We found that the force constants for the octahedral site complexes are low in comparison to that of the tetrahedral sites. However, both the force constants increase with increasing Zn content. Furthermore, the longitudinal elastic wave velocities are higher than the transverse wave velocities. The elastic moduli such as Young's modulus ( $E$ ), bulk modulus ( $B$ )

and rigidity modulus ( $G$ ) showed increasing trends with Zn concentration in the samples. The calculated Debye temperature ( $\theta_D$ ) is close to the experimentally obtained values. Our results provide a better understanding of the variation of mechanical, elastic and thermal properties of Li-Zn ferrites. The structural, elastic and thermal properties of all synthesized samples were studied in detail [26].

### Annexure VIII-3

#### Conclusion:

The main findings of the present work are given in the following;

The main objective of the present work was to synthesize the Li-Zn ferrite from a low temperature combustion synthesis technique by using Polyethylene Glycol (PEG) of molecular weight 6000 as fuel and to study the structural, elastic & thermal, magnetic and electrical properties of Li-Zn ferrites.

Zn- substituted lithium ferrite samples ( $\text{Li}_{10.5-x/2}\text{Zn}_x\text{Fe}_{2.5-x/2}\text{O}_4$ , where  $x = 0.0, 0.2, 0.4, 0.6, 0.8$  and  $1.0$ ) were prepared by combustion synthesis technique. The Li-Zn ferrite particles have rod shaped structure. Rietveld analysis of XRD data showed the structural transition from the  $\alpha$ -spinel (for pure lithium ferrite) to  $\beta$ -spinel on Zn-substitution. Also, with the increase in Zn-content, due to limited solid solubility, the impurity phases were found to have formed, whose abundance increased with Zn- content. The cation distributions at the interstitial sites confirmed negligible occupation of  $\text{Zn}^{2+}$  in the octahedral sites. FTIR spectroscopy corroborated the XRD results in confirming the cubic spinel phase formation with the absorption bands between  $\sim 550$ - $800 \text{ cm}^{-1}$  ( $\nu_1$ ) corresponding to tetrahedral- and between  $\sim 300$ - $500 \text{ cm}^{-1}$  ( $\nu_2$ ) corresponding to octahedral- complexes in the spinel structure of the Li-Zn ferrite. The results obtained from the XRD are well supported by the RT Mössbauer spectroscopy and magnetic measurements. Our

work demonstrates a cost effective way to synthesize Li-Zn ferrite samples by using simple combustion synthesis method.

The correlation between the tetrahedral and octahedral complexes and their relation to the splitting of the infrared absorption band of Zn- substituted lithium ferrite samples were studied by FTIR spectroscopy. The cubic spinel phase is confirmed via the observation of two absorption bands at  $\sim 510-800 \text{ cm}^{-1}$  ( $\nu_1$ ) corresponding to tetrahedral-complexes and at  $\sim 300-510 \text{ cm}^{-1}$  ( $\nu_2$ ) corresponding to octahedral-complexes in the spinel structure of the Li-Zn ferrite. The tetrahedral and octahedral bands split due to the presence of light weight  $\text{Li}^+$  ions at the octahedral site along with heavy  $\text{Fe}^{3+}$  ions. The band splitting observed in case of lithium ferrite disappeared with increasing Zn content which is interpreted as the increased disorder in cation distribution at octahedral sites. Our observation shows that the correlated vibrations of the tetrahedral and octahedral complexes decide the infrared absorption spectra of Li-Zn ferrites.

The elastic and thermal properties of Zn- substituted lithium ferrite samples were studied using FTIR spectroscopy. The tetrahedral and octahedral bands split due to the presence of light weight  $\text{Li}^+$  ions at the octahedral site along with heavy  $\text{Fe}^{3+}$  ions. The band splitting observed in case of lithium ferrite disappeared with increasing Zn content which is interpreted as the increased disorder in cation distribution at octahedral sites. The force constant for the tetrahedral site complexes are higher than that of the octahedral site complexes. The elastic sound waves travels faster longitudinally than that in the transverse direction. The elastic (Young's, Bulk and Rigidity) moduli calculated using the method by Waldron showed the progressive increase in their values with Zn-concentration in the samples. A comparison between Debye temperatures ( $\theta_D$ ) estimated by Waldron and Anderson methods showed that Anderson method resulted in lower value of  $\theta_D$  in comparison to the method by Waldron, but is more close to the reported

experimental value. The high elastic moduli and Debye temperature for our samples are due to stronger interaction between ions resulting in stiffer crystal.

The low coercivity values observed for all the samples suggest that the samples are soft magnetic in nature. As the non-magnetic Zn-content increases in the samples the saturation magnetization decreases and the remanent magnetization also decreases (making the LZF10 sample almost paramagnetic) at RT.

The sextet distribution signifies that there is a small disorder in the spatial positions of the  $\text{Li}^+$  ions (and hence,  $\text{Fe}^{3+}$  ions) present at the octahedral sites. The Mössbauer spectra for the LZF2 sample shows a sextet pattern with the Mössbauer lines tailing towards the lower hyperfine field values. This happens due to the substitution of non-magnetic  $\text{Zn}^{2+}$  ions at the tetrahedral site of the spinel structure of Li-ferrite

#### **Contribution to the society:**

Lithium-Zinc ferrites are low cost materials used for a wide range of technological applications such as in microwave circulators, isolators, phase shifters, sensors and other microwave applications.

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**List of publications and Conferences attended:**

**Conferences:(Presented Papers)**

- **Synthesis and morphology Studies of Lithium Zinc ferrite**  
*Vinodkumar Rathod, V M Jali and V.A Hiremath*  
25<sup>th</sup> AGM- Materials Research Society of India  
Indian Institute of Science, Bengaluru, Feb. 12 – 14, 2014.

**Publications:**

- **Combustion synthesis, structure and magnetic properties of Li-Zn ferrite ceramic powders**  
V. Rathod, Anupama A.V, V. M. Jali, V. A. Hiremath and B. Sahoo  
Ceramic International, 43 (2017) 14431-14440 (Publisher ELSEVIER)
- **Correlated vibrations of the tetrahedral and octahedral complexes and splitting of the absorption bands in FTIR spectra of Li-Zn ferrites**  
V. Rathod, Anupama A.V, R. Vijaya Kumar, V. M. Jali and B. Sahoo  
Vibrational Spectroscopy, 92 (2017) 267-272 (Publisher ELSEVIER)

  
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(Principal Investigator)

**Dr. Vinodkumar Rathod**

M.Sc. M.Phil. Ph.D.

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**PRINCIPAL**

Govt. College (Autonomous)

KALABURAGI-585102



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विश्वविद्यालय अनुदान आयोग

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PROVISIONAL APPROVAL LETTER

MBP(S)/13-14/KAGU009/UGC-SWRO

SHRI VINOD KUMAR RATHOD  
ASSISTANT PROFESSOR  
Dept. of PHYSICS  
GOVERNMENT COLLEGE  
SEDAM ROAD  
GULBARGA - 585 105

15-Feb-14

**Sub:** Financial Assistance to SHRI VINOD KUMAR RATHOD, GOVERNMENT COLLEGE, SEDAM ROAD, GULBARGA for undertaking Minor Research Project in Sciences for the project entitled SYNTHESIS AND CHARACTERIZATION OF LITHIUM-ZINC FERRITES.

Sir/Madam,

This is in reference to the proposal submitted by SHRI. VINOD KUMAR RATHOD, ASSISTANT PROFESSOR, to UGC for financial assistance under the above scheme. The proposal was placed before an Expert Committee duly constituted for the purpose.

Items	Amount Recommended
<b><u>Non-Recurring:</u></b>	
Books and Journals	10000/-
<b><u>Recurring :</u></b>	
Contingency	20000/-
Chemicals	40000/-
Fieldwork and Travel	5000/-
<b>TOTAL :</b>	<b>75000/-</b>

On receipt of the Approval letter, the Principal Investigator should inform the undersigned of his/her consent to implement the project and send the Acceptance Certificate(Annexure-I) before 10.3.14, otherwise it will be presumed that the Principal Investigator (PI) is not willing to implement the project and the approval will be withdrawn.

The grant is subject to the terms and conditions as per XIth plan guidelines on Minor Research Project and availability of funds. *and final approval by the UGC*

Yours faithfully

(Neethu S Phulaseedharan)  
Education Officer

Encl: As above

Copy to

1. The Principal  
GOVERNMENT COLLEGE  
SEDAM ROAD  
GULBARGA - 585 105

2. Office copy



विश्वविद्यालय अनुदान आयोग  
नेरुत्य प्रादेशिक कार्यालय  
**UNIVERSITY GRANTS COMMISSION**  
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P.K. Block, Palace Road, Gandhinagar  
Bangalore-560 009.  
Phone : (080) 2228 0380 Fax : (080) 2228 0381

MRP(S)-0425/13-14/KAGU009/UGC-SWRO

The Principal  
GOVERNMENT COLLEGE  
SEDAM ROAD  
GULBARGA - 585 105 .

08-Apr-14

**Sub: Transfer of funds to Colleges through RTGS/NEFT**

Sir/Madam,

This has reference to this office Sanction letter No. MRP(S)-0425/13-14/KAGU009/UGC-SWRO dated 28-Mar-2014 sanctioning therewith an amount of Rs.42500/- under the scheme of Minor Research Project

The above sanctioned amount has been transferred to your college Account No.0513101060635 with IFSC code: CNRB0000513 through RTGS/ NEFT.

The CANARA BANK, CUNNINGHAM ROAD, BANGALORE (CNRB0000431) has confirmed the above transfer of funds to your college through RTGS/NEFT vide confirmation number P14040781658311 on dated:08-Apr-2014

**You are requested to confirm the receipt of the above amount in your account by sending back the enclosed stamped receipt(colour paper). Further grants to the college will depend on receipt of this acknowledgement within ten days.**

Yours faithfully,

*N. G. Gopalky*  
Deputy Secretary

Encl. 1. Sanction order

2. Acknowledgement

*1/4*  
*23/4/14*

Diary No.

5811

MRP(S)-0425/13-14/KAGU009/UGC-SWRO  
The Accounts OfficerSouth Western Regional Office  
University Grants Commission  
P.K. Block, Palace Road  
Gandhinagar, Bangaloreविश्वविद्यालय अनुदान आयोग  
नैरुत्य प्रादेशिक कार्यालय  
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P.K. Block, Palace Road, Gandhinagar  
Bangalore-560 009.  
Phone : (080) 2228 0380 Fax : (080) 2228 0381  
28-Mar-14

Sub: Release of Grants-in-aid to GOVERNMENT COLLEGE, SEDAM ROAD, GULBARGA for the year 2013-2014 under MRP(S) (Plan) entitled SYNTHESIS AND CHARACTERIZATION OF LITHIUM-ZINC FERRITES.

Sir/Madam,

I am directed to convey the sanction of the University Grants Commission for payment of grant of Rs.42500/- as first installment for the year 2013-2014 to GOVERNMENT COLLEGE, SEDAM ROAD, GULBARGA under Minor Research Project (Plan) to Principal Investigator VINOD KUMAR RATHOD expenditure to be incurred during 2013-2014.

Items	Amount Allocated Rs.	HEAD OF ACCOUNT	Grant now being Sactioned	Grant already Sactioned	Total Grant	Balance
<b>Non-Recurring:</b>						
Books and Journals	10000/-	5(viii)	10000/-	0	10000/-	0
<b>Recurring :</b>						
Contingency including special needs	20000/-		10000/-	0	10000/-	10000/-
Chemicals and Glassware	40000/-		20000/-	0	20000/-	20000/-
Fieldwork and Travel	5000/-		2500/-	0	2500/-	2500/-
<b>Total :</b>	<b>75000/-</b>		<b>42500</b>	<b>0</b>	<b>42500</b>	<b>32500/-</b>

- The sanctioned amount is debitable to 5(viii) and is valid for payment during the financial year 2013-2014 only.
- The amount of the Grant shall be drawn by the Accounts Officer/Drawing and Disbursing Officer, South Western Regional Office, UGC, Bangalore on the Grants-in-aid bill and shall be disbursed to and credited to the Principal of the College through Electronic mode as per the following details:
  - Details (Name & Address) of Account Holder: GOVERNMENT COLLEGE, SEDAM ROAD, GULBARGA
  - Account No: 0513101060635
  - Name & address of Bank branch: CANARA BANK, . .
  - MICR Code: 585015002
  - IFSC Code: CNRB0000513
  - Type of Account: SB
- The grant is subject to the adjustment on the basis of utilisation certificate in the prescribed proforma submitted by the College.
- The college shall maintain proper accounts of the expenditure out of the grants which shall be utilised only on approved items of expenditure.
- The College may follow the General Financial Rules, 2005 and take urgent necessary action to amend their manuals of financial procedures to bring them in conformity with GFRs, 2005 and those don't have their own approved manuals on financial procedures may adopt the provisions of GFRs, 2005 and instructions/guideline there under from time to time.
- The Utilization Certificate to the effect that the grant has been utilized for the purpose for which it has been sanctioned shall be furnished to UGC as early as possible after the close of current financial year.

Contd.2

8. The assets acquired wholly or substantially out of University Grants Commission's Grant shall not be disposed or encumbered or utilised for the purposes other than those for which the grant was given without proper sanction of the UGC and should at any time the College ceased to function, such assets shall revert to the University Grants Commission.
9. A Register of Assets acquired wholly or substantially out of the grants shall be maintained by the College in the prescribed proforma.
10. The grantee institution shall ensure the Utilization of grants-in-aid for which it is being sanctioned/paid. In case of non-utilization/part utilization thereof, the simple interest @ 10% per annum as amended from time to time on unutilised amount from the date of drawal to the date of refund as per provisions contained in General Financial Rules of Govt. of India will be charged.
11. The College shall follow strictly the Government of India/UGC's guidelines regarding implementation of the reservation of policy [both vertical (for SC, ST and OBC) and horizontal ( for person with disability etc.]] in teaching and non-teaching posts.
12. The College shall fully implement the Official Language Policy of Union Govt. and comply with the Official Language Act, 1963, and Official Languages (Use for Official Purposes of the Union) Rules, 1978 etc.
13. The sanction is issued in exercise of the delegation of powers vide UGC office order No.130/2013[F.No.10-11/12(Admn. IA B)] dated 28/5/2013.
14. The College shall strictly follow the UGC Regulations on curbing the menace of Ragging in Higher Education Institutions, 2009.
15. The College shall take immediate action for its accreditation by National Assessment and Accreditation Council (NAAC).
16. The accounts of the College will be open for audit by the Comptroller and Auditor General of India in accordance with the provisions of General Financial Rules, 2005.
17. The annual accounts i.e. balance sheet, income and expenditure statement and statement of receipts and payments are to be prepared strictly in accordance with the Uniform Format of Accounting prescribed by the Government.
18. The funds to the extent are available under the Scheme.
19. This issues with the concurrence of IFD and approval of Secretary vide Diary No. 7900 dated 06-Mar-2014 respectively.
20. An amount of Rs nil out of the grant of Rs nil sanctioned vide letter No.MRP(S)-0425/13-14/KAGU009/UGC-SWRO dated nil has been utilized by the College for the purpose for which it was sanctioned and noted in Grants-in-aid Register at Page No. \_\_\_\_
21. The grant is sanctioned on the basis of the information/documents provided by the college. In case of any discrepancy in the above information and the college is found ineligible for the above grant at the time of expert committee meeting the college is liable to refund the sanctioned grant along with interest.
22. The college shall ensure involvement of Technical advice on and supervision of specifications and construction standards.

Yours faithfully

(Dr.N. Gopukumar)

Deputy Secretary

Copy to

1. The Principal  
GOVERNMENT COLLEGE  
SEDAM ROAD  
GULBARGA - 585 105  
(He/She is requested to abide by these instructions/guidelines of sanction order. )
2. SHRI VINOD KUMAR RATHOD  
ASSISTANT PROFESSOR  
GOVERNMENT COLLEGE  
SEDAM ROAD  
GULBARGA - 585 105
3. Officer of Director General of Audit, Central Revenues, AGCR Building, I.P. Estate, New Delhi
4. The Commissioner  
Department of Collegiate Education  
Government of Karnataka, Bangalore -
5. The Dean/Director, College Development Council  
GULBARGA UNIVERSITY  
GNANA GANGA  
GULBARGA - 585 106

6. Office copy

**NEETHU S. THULASEEDHAR**  
Education Officer  
University Grants Commission  
South Western Regional Office  
Palace Road, Gandhi Nagar  
BANGALORE - 560 002

**ACKNOWLEDGEMENT FOR THE GRANT RECEIVED FROM UGC-SWRO, BANGALORE BY RTGS/NEFT**  
( TO BE RETURNED IMMEDIATELY ON RECEIPT OF THE GRANT)

To  
The Regional Head  
University Grants Commission  
South Western Regional Office  
Prasanna Kumar Block  
Palace Road, Bangalore - 560 009

Received from University Grants Commission, South Western Regional Office, Bangalore a grant of

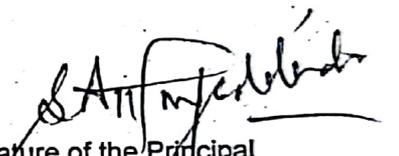
Rs: 42,500/- (Rupees Forty two thousand five hundred only)

RTGS/NEFT Number P14040781658311 dated 08-04-2014

in terms of sanction number MRP(CS)-0425/13-14/KAGU009 | UGC-SWRO dated 28-3-2014

for the purpose of Research.

Date: 25.6.2014  
Station: Gulbarga.

  
Signature of the Principal  
Full Name SAYEED AHMED  
Office Seal MUJADDADI  
Govt. College, Gulbarga

NOTE: FURTHER GRANT WILL BE RELEASED ONLY ON RECEIPT OF THE ABOVE ACKNOWLEDGEMENT DULY SIGNED BY THE PRINCIPAL WITHIN 10 DAYS FROM THE RECEIPT OF THE GRANT.



# Materials Research Society of India (MRSI)

14<sup>th</sup> February 2014

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# Combustion synthesis, structure and magnetic properties of Li-Zn ferrite ceramic powders

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## ABSTRACT

We report the effect of Zn-substitution on the structure and magnetic properties of Li-ferrite powders ( $\text{Li}_{0.5-x}/2\text{Zn}_x\text{Fe}_{2.5-x}/2\text{O}_4$ ,  $x = 0.0, 0.2, 0.4, 0.6, 0.8$  and  $1.0$ ) synthesized by combustion synthesis route. Different stages involved during progress of combustion synthesis of Li-Zn ferrite samples were monitored by thermogravimetric analysis (TGA) and differential scanning calorimetry (DSC). The scanning electron micrographs of the synthesized samples show foamy network type of morphology with rod-shaped particles. Rietveld refinement of the X-ray diffraction (XRD) data confirmed the preferential occupancy of the Zn-atoms at the tetrahedral sites and the Li atoms at the octahedral sites of the spinel structure. Trace amounts of zinc-oxide and iron-oxide impurity phases were found for the samples with high Zn content ( $x \geq 0.6$ ). The comparison of the particle sizes obtained from scanning electron micrographs and the crystallite sizes estimated from XRD results suggest that the particles are polycrystalline aggregates of a few crystalline grains. Fourier transform infrared (FTIR) spectroscopy confirmed the presence of broad (vibrational) absorption bands between  $\sim 510$  and  $800 \text{ cm}^{-1}$  ( $\nu_1$ ) corresponding to tetrahedral complexes and between  $\sim 250$  and  $510 \text{ cm}^{-1}$  ( $\nu_2$ ) corresponding to octahedral complexes in the spinel structure of the Li-Zn ferrites. The phase compositions of the samples were obtained from the analysis of Mössbauer spectra and XRD patterns. Substitution of higher amount of  $\text{Zn}^{2+}$  ions reduces the saturation magnetization of the samples. Our work demonstrates that combustion synthesis method is a cost effective and simple way to synthesize bulk amount of Li-Zn ferrite samples with desired physical properties.

## 1. Introduction

Pure and substituted lithium ferrites are low cost materials used for a wide variety of technological applications [1–6]. Lithium ferrite finds applications in many high frequency electronic devices such as in microwave circulators, isolators, phase shifters and absorbers, because of its high permeability (in microwave frequency range) and high Curie temperature. The other properties which make Li-ferrite suitable for such applications are its high electrical resistivity, mechanical strength and chemical stability [2,7]. Lithium ferrite is often doped with other cations to optimize its electrical, magnetic and elastic properties [2]. Previously, non-magnetic ion substituted lithium ferrites were prepared using different methods such as conventional double sintering [8], sol-gel [9–11] and solid-state reaction of inorganic precursors [12] etc. Synthesis of lithium ferrites using these methods is either time and

power consuming or cumbersome. In addition, in solid state reaction route, high calcination temperature lowers the magnetization due to precipitation of  $\alpha\text{-Fe}_2\text{O}_3$  or formation of  $\text{Fe}_3\text{O}_4$  phase along with a non-magnetic phase (e.g., ZnO in case of Li-Zn ferrites). Furthermore, the reduction of  $\text{Fe}^{3+}$  to  $\text{Fe}^{2+}$  (during the formation of  $\text{Fe}_3\text{O}_4$ ) can lead to an increased electrical conductivity limiting these materials in microwave applications, where high resistivity and minimum dielectric loss are required [7].

Previously, Li-Zn ferrite powders were prepared by simple methods such as microwave-induced glycine-nitrate process [13,14] and solution combustion [15]. These methods require nitrate precursors which are hygroscopic, not always commercially available and often expensive than oxide precursors. To overcome these limitations, we have used 'combustion synthesis' method [16,17], which is an advanced approach in powder metallurgy. In this method, the oxide precursors (solids) and

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# Correlated vibrations of the tetrahedral and octahedral complexes and splitting of the absorption bands in FTIR spectra of Li-Zn ferrites

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## ABSTRACT

We report the infrared absorption properties of Li-Zn ferrite ( $\text{Li}_{0.5-x/2}\text{Zn}_x\text{Fe}_{2.5-x/2}\text{O}_4$ ,  $x = 0.0, 0.2, 0.4, 0.6, 0.8$  and  $1.0$ ) powder samples prepared by combustion synthesis. The FTIR spectrum for each of the sample consists of two broad absorption bands: one at  $\sim 510\text{--}800\text{ cm}^{-1}$  corresponding to the vibrations of tetrahedral complexes ( $\nu_1$ ) and the other, at  $\sim 300\text{--}510\text{ cm}^{-1}$  corresponding to the vibrations of octahedral-complexes ( $\nu_2$ ) in the spinel structure of the Li-Zn ferrites. Each of these bands is further split into four sub-bands. We have explained the observed infrared absorption bands based on the structure and the occupancy of the cations. Pronounced splitting of the  $\nu_1$  and  $\nu_2$  bands in pure  $\text{Li}_{0.5}\text{Fe}_{2.5}\text{O}_4$  were observed due to the  $\text{LiO}_6$  complexes partially replacing the  $\text{FeO}_6$  complexes at the octahedral interstitial sites. The vibrational frequencies of the  $\text{LiO}_6$  complexes are different from  $\text{FeO}_6$  complexes due to the difference in their reduced mass and valence of the cations (lower bond strength). The resolved bands smear out with increasing Zn content ( $x$ ) owing to higher statistical disorder in the occupancy of the cations at the tetrahedral and octahedral sites. We observe that the vibrations of the tetrahedral and octahedral complexes are not isolated but rather correlated. Our work helps in understanding the correlation of cation occupancy with the vibrational properties of nanocrystalline Li-Zn ferrites.

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## 1. Introduction

Lithium ferrites are low cost materials used for a wide variety of technological applications [1–6]. Lithium ferrites are used in many electronic devices for high frequency applications such as in microwave circulators, isolators, phase shifters and absorbers, due to their high permeability in this frequency range and high Curie temperature. The other properties which make Li-ferrite suitable for such applications are its high electrical resistivity, mechanical strength and chemical stability [2,7]. Lithium ferrites are spinel ferrites having general formula  $\text{AB}_2\text{O}_4$ . For spinels this formula is generally represented as  $(\text{A})[\text{B}_2]\text{O}_4$ , where the parenthesis represents the tetrahedral site and the square bracket represents the octahedral site. In normal spinel ferrites the tetrahedral (A-) site is occupied by a divalent cation (such as  $\text{Zn}^{2+}$ ,  $\text{Mg}^{2+}$  etc.) and octahedral (B-) site is occupied by trivalent cation (such as  $\text{Fe}^{3+}$ ). In

inverse spinel ferrites, the divalent cations (such as  $\text{Ni}^{2+}$ ) occupy the B site. In some ferrites, the occupation of divalent cations is distributed in A- and B-sites: these ferrites are known as mixed spinel ferrites. Our studied samples, Zn-doped lithium-ferrites, are typical examples of mixed spinel ferrites of the type  $(\text{Zn}_x\text{Fe}_{1-x})[(\text{Li}_{1-x/2}\text{Fe}_{(1-x)/2})\text{Fe}_{(1+x)}]\text{O}_4$ , where the  $\text{Zn}^{2+}$  cation goes to the A-site and the combination of cations,  $(\text{Li}_{0.5}\text{Fe}_{3+0.5})$ , which forms the divalent-metal cation, goes to the octahedral (B-) sites. The rest of the  $\text{Fe}^{3+}$  cations are distributed in the A- and B-sites. The distribution of different cations at the interstitial sites modifies the infrared absorption spectrum, as the dipole moment change during vibration of the cation complexes varies due to different mass, charge and strength of interaction between A- and B-site complexes.

The mechanical properties of materials can be understood from the nature of the chemical bond and their vibrational properties. In general, most of the properties such as mechanical, vibrational, elastic and thermal etc. depend on the nature of binding forces between the atoms in the materials. As the binding forces depend on the type of atoms/ions and the inter-atomic/inter-ionic forces involved, the substitution of host element by dopants invariably

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