



### 3. Electrochemistry

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#### Abstract

Understanding of electrical matters began within the sixteenth century. Throughout this century, land somebody William Gilbert spent seventeen years experiment with magnetism and, to a lesser extent, electricity. For his work on magnets, Gilbert became referred to as the "Father of Magnetism." He exposed varied ways for manufacturing and strengthening magnets.

Electrochemistry is that the branch of chemical science that studies the connection between electricity, as a measurable and quantitative development, and recognizable action, with either electricity thought-about Associate in outcome of a selected action or the other way around. These reactions involve electrical charges moving between electrodes Associate in solution (or ionic species in an exceedingly solution). Therefore chemistry deals with the interaction between current and action.

#### Introduction

When a chemical change is caused by Associate in Nursing outwardly equipped current, as in electrolysis, or if an electrical current is created by an unprepared chemical change as in an exceedingly battery, it's referred to as Associate in Nursing chemistry reaction. Chemical reactions wherever electrons area unit transferred directly between molecules and/or atoms area unit referred to as oxidation-reduction or (redox) reactions. In general, chemistry describes the reactions once individual reaction reactions area unit separate however connected by Associate in peripheral electrical circuit Associate in an intervening solution.

The term "redox" stands for reduction-oxidation. It refers to chemistry processes involving lepton transfer to or from a molecule or particle dynamical its number. This reaction will occur from starting to finish the appliance of Associate in nursing external voltage or through the discharge of energy. Reaction and reduction describe the modification of number that takes place within the atoms, ions or molecules concerned in Associate in nursing chemistry reaction. Formally, number is that the hypothetical charge that Associate in nursing atom would have if all bonds to atoms of various components were one hundred ionic. Associate in Nursing atom or particle that provides up Associate in nursing lepton to a different atom or particle has its number increase, and therefore the beneficiary of the charged lepton has its number diminish.





For example, once atomic Na reacts with atomic Cl, Na donates one lepton Associate in Nursing attains a number of +1. Cl accepts the lepton and its corrosion state are reduced to -1. The sign of the number (positive/negative) really corresponds to the worth of every ion's electronic charge. The attraction of the otherwise charged Na Associate in Nursing Cl ions is that the reason they then type an electrostatic bond.

The loss of electrons from Associate in Nursing atom or molecule is named reaction, and therefore the gain of electrons is reduction. This may be simply remembered through the employment of method devices. 2 of the foremost well-liked area unit "OIL RIG" (Oxidation Is Loss, Reduction Is Gain) and "LEO" the lion says "GER" (Lose Electrons: reaction, Gain Electrons: Reduction). Reaction and reduction perpetually occur in an exceedingly paired fashion such one species is altering once an extra is reduced. For cases wherever electrons area unit shared (covalent bonds) between atoms with massive variations in electro negativity, the lepton is allotted to the atom with the most important electro negativity in decisive the number.

The atom or molecule that loses electrons is thought because the reducer, or chemical agent, and therefore the substance that accepts the electrons are named the oxidiser, or oxidant. Thus, the oxidiser is usually being reduced in an exceedingly reaction; the reducer is usually being alter. O may be a common oxidiser, however not the sole one. Despite the name, Associate in Nursinging reaction reaction doesn't essentially have to involve O. In fact, a fireplace may be fed by Associate in Nursinging oxidizer although oxygen; halogen fires area unit often insatiable, as halogen is a good stronger oxidizer (it encompasses a higher electro negativity and therefore accepts electrons even better) than O.

For reactions involving O, the gain of O implies the reaction of the atom or molecule to that the O is other (and the O is reduced). In organic compounds, comparable to alkane or alcohol, the loss of H implies reaction of the molecule from that it's lost (and the H is reduced). This follows as a result of the H donates its lepton in valency bonds with non-metals however it takes the lepton on once it's lost. Conversely, loss of O or gains of H imply reduction.

### Electrochemical Cells

A chemistry cell may be a device that produces an electrical current from energy free by a spontaneous reaction; this may be caused from electricity. This sort of cell includes the cell or electric cell, named when Galvani and Alessandro Volta, each scientists UN agency conducted many experiments on chemical reactions and electrical phenomenon throughout the late eighteenth century.





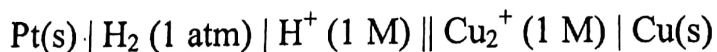
The SHE conductor may be associated to the other conductor by a salt bridge to make a cell. If the second conductor is additionally at normal conditions, then the measured cell potential is named the quality conductor potential for the conductor. The quality conductor potential for the SHE is zero, by classification. The polarity of the quality conductor potential provides info concerning the relative reduction potential of the conductor compared to the SHE. If the conductor encompasses a positive potential with relation to the SHE, then which means it's a powerfully reducing conductor that forces the SHE to be the anode (an example is cubature unit liquid  $\text{CuSO}_4$  with a regular conductor potential of zero.337 V). Conversely, if the measured potential is negative, the conductor is a lot of oxidizing than the SHE (such as Zn in  $\text{ZnSO}_4$  wherever the conventional conductor potential is  $-0.76$  V).

Standard conductor potentials area unit sometimes tabulated as reduction potentials. However, the reactions area unit reversible and therefore the role of a selected conductor in an exceedingly cell depends on the relative oxidation/reduction potential of each electrodes. The reaction potential for a selected conductor is simply the negative of the reduction potential. A regular cell potential may be determined by wanting up the quality conductor potentials for each electrodes (sometimes referred to as [\*fr1] cell potentials). The one that's smaller are the anode and can bear reaction. The cell potential is then calculated because the addition of the reduction potential for the cathode and therefore the reaction potential for the anode.

$$E^\circ_{\text{cell}} = E^\circ_{\text{red}} (\text{cathode}) - E^\circ_{\text{red}} (\text{anode}) = E^\circ_{\text{red}} (\text{cathode}) + E^\circ_{\text{oxi}} (\text{anode})$$

For example, the quality conductor potential for a copper conductor is:

Cell diagram



$$E^\circ_{\text{cell}} = E^\circ_{\text{red}} (\text{cathode}) - E^\circ_{\text{red}} (\text{anode})$$

At C, pressure and concentration conditions, the cell's voltage (measured by a multimeter) is zero.34 V. By definition, the conductor potential for the SHE is zero. Thus, the Cu is that the cathode and therefore the SHE is that the anode giving

$$E_{\text{cell}} = E^\circ(\text{Cu}_2^+/\text{Cu}) - E^\circ(\text{H}^+/\text{H}_2)$$

Or,

$$E^\circ(\text{Cu}_2^+/\text{Cu}) = \text{zero.34 V}$$

Changes within the ratio coefficients of a balanced cell equation won't modification  $E^\circ_{\text{red}}$  worth as a result of the quality conductor potential is Associate in nursing intensive property.



## Battery

Many types of battery are commercial and represent a crucial use of chemistry. Early wet cells battery-powered the primary telegraphs and phone systems, and were the supply of current for electroplating. The zinc-manganese oxide Leclanche cell was the primary transportable, non-spillable battery kind that created flashlights and alternative transportable devices sensible. The mercury battery exploitation atomic number 30 and metal chemical compound provided higher levels of power and capability than the innovative Leclanche cell for early electronic devices. however has been phased out of common use thanks to the danger of mercury pollution from discarded cells.

The lead-acid battery was the primary smart secondary (rechargeable) battery that would have its capability replenished from Associate in nursing external supply. The chemistry reaction that created current was (to a helpful degree) reversible, permitting current and energy to be interchanged PRN. Common lead acid batteries contain a combination of acid and water, also as lead plates. The foremost common mixture used these days is half-hour acid. One downside but is that if left uncharged acid can crystallize inside the lead plates of the series rendering it useless. These batteries last a mean of three years with daily use but it's not exceptional for a lead acid battery to still be purposeful when 7-10 years. Lead-acid cells still are wide utilized in vehicles.

All the preceding varieties have water-based electrolytes that limit the most voltage per cell. The state change of water limits temperature performance. The metal battery, that doesn't (and cannot) use water within the solution, provides improved performance over alternative types; a chargeable lithium-ion battery is a vital a part of several mobile devices.

The flow battery, Associate in nursing experimental kind, offers the choice of immensely larger energy capability as a result of its reactants may be replenished from external reservoirs. The cell will flip the energy sure in organic compound gases or H directly into current with abundant higher potency than any combustion process; such devices have battery-powered several satellite and area unit being applied to grid energy storage for the general public power grid.

## Corrosion

Corrosion is Associate in nursing chemistry method that reveals itself in rust or tarnish on metals like iron or copper and their various alloys, steel and brass.



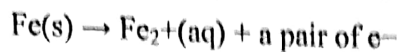




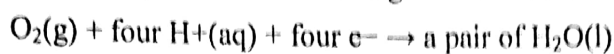
## Iron Corrosion

For iron rust to occur the metal should be involved with O and water, though chemical reactions for this method area unit comparatively complicated and not all of them area unit fully understood. It's believed the causes area unit the following: lepton transfer (reduction-oxidation)

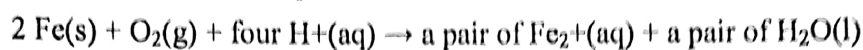
One space on the surface of the metal acts because the anode, that is wherever the reaction (corrosion) happens. At the anode, the metal provides up electrons.



Electrons area unit transferred from iron, reducing O within the atmosphere into water on the cathode that is placed in another region of the metal.



Global reaction for the process



Standard voltage for iron rusting

$$E^\circ = E^\circ(\text{cathode}) - E^\circ(\text{anode})$$

$$E^\circ = 1.23 \text{ V} - (-0.44 \text{ V}) = 1.67 \text{ V}$$

An electric circuit is created as passage of electrons and ions happens, therefore if Associate in nursing solution is gift it'll facilitate reaction, explaining why corroding is faster in salt water.

## Corrosion of Common Metals

Coinage metals, comparable to copper and silver, slowly corrode through use. A coat of green-blue copper carbonate forms on the surface of copper with exposure to the water and CO<sub>2</sub> within the air. Silver coins or cutlery that area unit exposed to high sulfur foods comparable to eggs or the low levels of sulfur species within the air develop a layer of black Silver chemical compound.

Gold and atomic number 78 is unit extraordinarily troublesome to oxidize beneath traditional circumstances, and need exposure to a robust chemical oxidiser comparable to nitrohydrochloric acid.

Some common metals oxidize extraordinarily speedily in air. Metallic element and metallic element oxidize outright involved with the O within the air. These metals type a particularly skinny layer of alter metal on the surface that bonds with the underlying metal. This skinny layer of chemical compound protects the underlying layers of the metal from the air preventing the complete metal from oxidizing. These metals area unit utilized in applications wherever corrosion resistance is vital. Iron, in distinction, has Associate in nursing chemical





compound that forms in air and water, referred to as rust, that doesn't bond with the iron and thus doesn't stop the any reaction of the iron. Therefore iron left exposed to air and water can still rust till all of the iron is oxidized.

### **Prevention of Corrosion**

Attempts to save lots of a metal from changing into anodal area unit of 2 general varieties. anodal regions dissolve and destroy the structural integrity of the metal.

While it's nearly not possible to forestall anode/cathode formation, if a non-conducting material covers the metal, contact with the solution isn't doable and corrosion won't occur.

### **Coating**

Metals may be coated with paint or alternative less semiconductive metals (passivation). This prevents the metal surface from being exposed to electrolytes. Scratches exposing the metal substrate can end in corrosion. The region beneath the coating adjacent to the scratch acts because the anode of the reaction.

### **Sacrificial Anodes**

A method ordinarily wont to defend a structural metal is to connect a metal that is a lot of anodal than the metal to be protected. This forces the structural metal to be cathodic, therefore spared corrosion. It is referred to as "sacrificial" as a result of the anode dissolves and should get replaced sporadically.

Zinc bars area unit hooked up to varied locations on steel ship hulls to render the ship hull cathodic. The atomic number 30 bars area unit replaced sporadically. Alternative metals, comparable to metal, would work alright however atomic number 30 is that the least dear helpful metal.

To protect pipelines, Associate in nursing metal bar of buried or exposed metal (or zinc) is buried beside the pipeline and is connected electrically to the pipe on top of ground. The pipeline is forced to be a cathode and is shielded from being alter and corroding. The metal anode is sacrificed. At intervals new ingots area unit buried to interchange those lost.

### **Electrolysis**

The spontaneous reaction reactions of a traditional battery manufacture electricity through the various chemical potentials of the cathode and anode within the solution. However, electrolysis needs Associate in Nursing external supply of current to induce a chemical change. Associate in Nursing this method takes place in an exceedingly compartment referred to as an electric cell.







### Electrolysis of Liquified Common Salt

When liquified, the salt common salt may be electrolyzed to yield aluminiferous Na and volatilized Cl. Industrially this method takes place in an exceedingly special cell named Down's cell. The cell is connected to Associate in nursing wattage offer, permitting electrons to migrate from the facility offer to the electric cell.

Reactions that occur at Down's cell area unit the following

Anode (oxidation): a pair of  $\text{Cl}^- \rightarrow \text{Cl}_{2(g)} + \text{a pair of } e^-$

Cathode (reduction): a pair of  $\text{Na}^{(l)} + \text{a pair of } e^- \rightarrow \text{a pair of } \text{Na}^{(l)}$

Overall reaction: a pair of  $\text{Na}^+ + \text{a pair of } \text{Cl}^{-(l)} \rightarrow \text{a pair of } \text{Na}^{(l)} + \text{Cl}_{2(g)}$

This method will yield massive amounts of aluminiferous Na and volatilised Cl, and is wide used on ore dressing and scientific discipline industries.

The voltage for this method is more or less  $-4 \text{ V}$  indicating a (very) non-spontaneous method. So as for this reaction to occur the facility offer ought to give a minimum of a possible of four V. However, larger voltages should be used for this reaction to occur at a high rate.

### Electrolysis of Water

Water may be regenerate to its part elemental gasses,  $\text{H}_2$  Associate in Nursing  $\text{O}_2$  through the appliance of an external voltage. Water does not decompose into H and O impromptu because the chemist free energy for the method at normal conditions is concerning  $474.4 \text{ kJ}$ . The decomposition of water into H Associate in Nursing O may be performed in an electric cell. In it, a try of inert electrodes sometimes manufactured from atomic number 78 immersed in water act as anode and cathode within the electrolytic method. The electrolysis starts with the appliance of Associate in nursing external voltage between the electrodes. This method won't occur except at extraordinarily high voltages while not Associate in nursing solution comparable to common salt or acid (most used zero.1 M).

Bubbles from the gases are seen close to each electrode. The subsequent [\*fr1] reactions describe the method mentioned above:

Anode (oxidation): a pair of  $\text{H}_2\text{O}(l) \rightarrow \text{O}_2(g) + \text{four } \text{H}^+(aq) + \text{four } e^-$

Cathode (reduction): a pair of  $\text{H}_2\text{O}(g) + \text{a pair of } e^- \rightarrow \text{H}_2(g) + \text{a pair of } \text{OH}^-(aq)$

Overall reaction: a pair of  $\text{H}_2\text{O}(l) \rightarrow \text{a pair of } \text{H}_2(g) + \text{O}_2(g)$

Although sturdy acids could also be utilized in the equipment, the reaction won't web consume the acid, whereas this reaction can work on any semiconductive conductor at a sufficiently massive potential, atomic number 78 catalyzes each H and O formation, granting comparatively delicate voltages ( $\sim 2 \text{ V}$  looking on the pH).



### Electrolysis of Liquid Solutions

Electrolysis in Associate in nursing liquid may be a similar method as mentioned in electrolysis of water. However, it's thought-about to be a fancy method as a result of the contents in resolution have to be compelled to be analyzed in [\*fr1] reactions, whether or not reduced or alter.

### Electrolysis of an Answer of Common Salt

The presence of water in an exceedingly resolution of common salt should be examined in relation to its reduction and reaction in each electrodes. Usually, water is electrolysed as mentioned in electrolysis of water yielding volatilised O within the anode and volatilised H within the cathode. On the opposite hand, common salt in water dissociates in  $\text{Na}^+$  and  $\text{Cl}^-$  ions, cation, that is that the positive particle, are interested in the cathode (-), therefore reducing the Na particle. The ion can then be interested in the anode (+) oxidizing chloride particle.

### Conclusion

There are unit varied extraordinarily necessary chemistry processes in each nature and business, just like the coating of objects with metals or metal oxides through deposition and therefore the detection of alcohol in boozy drivers through the reaction of alcohol. The generation of energy through photosynthesis is inherently associate in nursing chemistry process, as is production of metals like atomic number 13 and metallic element from their ores. Sure polygenic disorder aldohexose meters live the number of glucose within the blood through its reaction potential. also because the established chemistry technologies (like deep cycle lead acid batteries) there's conjointly a good vary of latest rising technologies comparable to fuel cells, massive format lithium-ion batteries, chemistry reactors and super-capacitors that have become progressively business. Chemistry has conjointly necessary applications within the food business, just like the assessment of food/package interactions, the analysis of milk composition, the characterization and therefore the determination of the state change end-point of ice-cream mixes, the determination of free acidity in oil. The action potentials that travel down connected neurons area unit supported electrical phenomenon generated by the movement of Na and K ions into and out of cells. Specialized cells in sure animals just like the electric eel will generate electric currents powerful enough to disable abundant larger animals.

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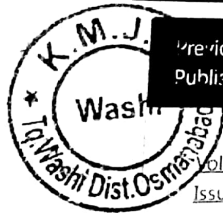




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## WEEDING POLICY OF ACADEMIC LIBRARIES IN INDIA

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*Abstract: This paper discusses in detail the weeding policy and what factors are useful in removing old collections/items from the library to keep the collection intact and to avoid gap problems while accommodating new collections.*

*KeyWords: CollectionDevelopment, Weeding Policy, Academic Libraries etc.*

### Introduction:

Weeding (De-selection) is an essential component of collection development who ensures that library materials are useful and accessible each library has limited space to available the store its collection. Also, the collection should change over time to reflect changes in society. Delisting is the continuous evaluation of resources, aimed at removing items or collections from the collection that are no longer useful. De-cluttering (redundant collection) of documents and other materials is considered an important function of libraries, so that they can be discarded and their collection updated.

The following steps are suggested for removing redundant or unwanted materials from the library. 1) Arrange such books in proper order, 2) Compile necessary relevant catalog records, 3) Maintain disposition slips in books for de-listing or de-listing, 4) Prepare tabular records for books drawn/de-delisted etc.

In such a case, prior approval of the concerned parent body i.e. Library Advisory Committee is required before effective registration.

### Meaning of Weeding:

Weeding is the systematic removal of resources from a library based on selected criteria. Content selection is the opposite, although content selection and non-selection often involve similar thought processes. Weeding is an important process for active collections in libraries

Ensures that the collection remains relevant and in good condition. Weeding should be done continuously, constantly. This will keep the quality of the collection good. Staff should be educated with periodic workshops and presentations on the importance and positive benefits of maintenance and collection weeding.

### **Requirement of weeding for Academic Library:**

Certain types of library materials become outdated with time. These outdated and unused books incur huge maintenance costs for libraries and slow down or hinder the workflow of library management in many ways, hence the need to dispose of these materials.

- Shortage of shelf-space every library, small or large, has limited shelf space and so to make best use of the available space, it is necessary to delist or remove books to make room for new books and future collections.
- De-duplication of materials: When a library has to acquire multiple copies of books. Also because of new syllabus changes, many copies of old books are left lying around in the library, when syllabus is changed or the latest editions are lost, so it is necessary to write them down.
- Are the collections relevant to the curriculum according to the student's needs? And books on other unrelated subjects are of no use to the student, while other books may be written off or eliminated, retaining one or two references. Increasing use of electronic resources through consortia or open access is gradually reducing the use of print in higher education institutions. Thus, duplicate print can either be set aside in a passive collection or removed if e-resources are purchased on a perpetual (perpetual access basis).
- Maintenance of books on open shelves (shelving), protection from insects, atmospheric humidity, binding or repair, archiving for posterity is constantly increasing and costs and wastes valuable human resources. Therefore, it is necessary to write down the unnecessary materials and keep the useful materials in the library.

### **Weeding Out Policy:**

#### **Meaning and purpose:**

Descriptive or material removal policy is an important component of collection development strategy. Removal of documents means removal of documents, books from the library which are not useful or serviceable. Documents removed from the library may be removed completely. Where they are unserviceable, others are donated to libraries where they may be useful; or kept in a reserved place called hostel library. Descriptive strategy means a) what material is to be described? b) When to write? c) Who should prescribe? d) How to write? e) What to do with the recorded material etc.

The current age is the age of knowledge and the explosion of knowledge is now bringing to light millions of new fields of knowledge and making some of the earlier ideas and thoughts obsolete. The explosion of knowledge has created a problem of money and space to purchase new materials. Which is relevant and useful material especially the space to store the documents has become a big problem for the libraries. Attempts have been made to find a solution for this earlier and even now efforts are underway to find a suitable solution. The only reason to consider decomposing is not to save space, but because constant use can cause certain texts or documents to become obsolete. Likewise, some documents may become brittle and unusable over time. But the most important reason is that some documents

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may become unusable because their content has been modified in a later version; or because the information in the document has become obsolete.

In modern times, books, other materials and documents can be removed. The information contained therein can be transferred to microforms and computer tapes. A report of the Library Committee of the University Grants Commission comments on the need for decommissioning: "Many works of art lose their value in a generation, less than twenty-five years. By then their ideological content may be out. It is wrong. To retain such pedantic books in service libraries and give them shelf space after they become obsolete." No useful purpose is served by giving it. The proper way is to write it down or remove it from time to time. They should accommodate their existing differences."

### **Discouraging Factors:**

Many librarians are reluctant or apathetic to implement library materials or collections that are motivated by compelling reasons to delist or remove them from time to time. It is very evident in our country. Some of the reasons for such depression are as follows

- Library numbers are very important in our country. The glory of numbers in the library continues today, although modern librarians realize that the quality of service is more important than the number of documents in a library, yet they are now reluctant to remove documents that are no longer useful. This is mainly because the official reports presented by them emphasize numbers.
- Sanctity of collection many people think that every book, however old it is, has more value because of its rarity. Sometimes they are of the opinion that even an accurate reprint of an antiquarian book increases its value, available in the market in good physical condition. But because of the time-consuming nature of transcription work in a service library, routine duties can be displaced.
- Fear of audit and customer comments: One may face during audit. Clients can also complain about misplaced materials or documents, just like a library that doesn't find documents for which money was paid for. Complaints of being censored from the library, claiming that some very useful documents have also been thrown away, are vexed. But the librarian should act without bias due to these factors and clear and well-planned writing policy approved by the library advisory committee appointed for the purpose or by the library committee.

### **What Materials are to be weeded out?**

Regarding the material to be written, J.S. Sharma (1978) listed the type of material to be withdrawn from the library as a subject of decommissioning policy. Also in 1994 H.R. Chopra recreated the same list. The following are some of the types of materials that need to be written down from time to time. They are said to be books that have been distorted by users due to constant use.

- Best-seller material, fictional non-fiction used by hundreds of readers, deteriorates after a while. Such books or documents should be registered.

- Textbooks and language books printed on poor quality paper deteriorate quickly. Therefore, books that cannot be bound once they are worn out should be written off.
- Science and technology are developing so rapidly that new research makes books published twenty or thirty years ago obsolete or out of date. Such books have to be scrapped and replaced with new editions or new books on the subject.
- Library reference books like yearbooks, yearbooks, handbooks etc. Those published from time to time should be replaced with new versions and old versions should be discarded to save space.
- Microforms, CD-ROM, Hard Disk etc. And with the advent of other space saving technologies useful information can be stored in them. Therefore some books and journals should be replaced by such new format documents after the frequency of use has decreased or at the very beginning
- Finally, the library should always keep in mind Ranganathan's fifth formula i.e. 'Library is a growing institution' As the collection grows or the number of documents increases, space becomes a problem, so all unnecessary materials must be removed to make room for new and urgently needed books and other documents.

### **When to weed out?**

During the day-to-day operations of the library, the librarian may at any time during the year come across books, texts or documents which are out of date.

Chopra Says, "In college and university libraries, every time the library handles a book, it should be checked from the angle of its physical condition and its continued suitability. The entire collection should be checked at least once a year." So what material has to be written down or constructed, if it is not useful then list it and write it down. An annual write-off (junk collection) from unwanted documents would be ideal.

### **Who should weed out?**

Extraction of literary material (collection) is an exercise that requires judgment and skill. The librarian has to be personally involved. The subject has to be proposed before the Library Advisory Committee at the university or college level, after which a resolution is passed in the University or College Development Committee (CDC) and a member of the Library Committee or a nominated member of the Library Authority as per the recommendation of that committee is appointed to re-examine the materials to be recorded or appointed for the purpose. The members of the Committee may take decisions and delist or withdraw materials from the library. For this, one has to consult subject experts or senior professors whose judgment can be trusted and if there is a university, the material has to be written.

### **How to weed out or Guidelines for Weeding out?**

Cataloging (redundant collection) materials in the library should not be arbitrary. Because once we remove a document or a document from the library, we should consider getting a new copy or a new edition or a new book on the subject in its place, and what if the

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same document is needed? A later date? Several theories and guidelines have emerged regarding waste disposal.

Simon felt that past usage is the best guide to future usage of texts or documents. Trueswell developed a technique for removing collections that would ensure future satisfaction of a given quantity by examining past usage. There are studies by Marianne Cooper and Raffael and ShishkoRaffael, and Shishko suggests that publication date should be an important criterion for classifying documents. The Council of the American Library Association believes that public libraries should "average at least 5 percent of total collections annually (unwanted collections)" stating that "unwanted items left in the collection can weaken the library.

The Sinha Committee Report (Library Advisory Committee Report) (1958) takes a similar view of Indian public libraries. The report found that "removal of worn and out-of-date books (redundant collections) is as important as the acquisition of new books in the public library. It is said that a conscientious librarian should remove 5 percent of his fiction and 2 percent of his literature.

### 1.1. What to do with Weeded out Materials :

What to do with Described or Removed Content? Of course, books and other materials can be sold just like old newspapers that are completely worn, damaged and irreplaceable. But if the materials or books are in good condition, especially if they are old editions, especially if they are old editions of reference sources like encyclopedias, handbooks etc., they have to be donated to other libraries which are not in a position to buy such expensive books.

Another way is to store documents that are otherwise useful, known as "book reservoirs". At least one copy of written or removed documents should be preserved in a place located on the territory of the country. Dr. S.R. Ranganathan chaired by: According to the UGC Committee on University and College Libraries (1965), "By extension, this is necessary in service libraries and generally modern university libraries are service libraries – they should not be done in a disjointed manner by libraries in the country. Therefore, for the use of posterity A few copies of every book should be preserved - and especially for archival and bibliographic research". Examination of documents should be done with specific knowledge and approval of the concerned library committee or library authority. In the Accession Register the document is recorded citing the relevant orders or proceedings of the concerned authority. Shelf lists, catalogs and other entries should be corrected by deleting entries relating to the removed documents

### Conclusion:

We conclude and review of all the above reveals that there are no prevailing standards or procedures for disposal of unused books in the library each library should formulate and document its own policy for material removal in consultation with subject matter experts, so that it forms policy guidelines for future operations. A detailed discussion on what content to

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delete, when to delete, who should delete and what to do with deleted/removed content etc. guidelines for how to catalog materials in libraries, i.e. opinion of Council of American Library Association, as well as Sinha Committee Report, etc. give impetus to cataloging.

Thus, the above detailed analysis, it is concluded that the complete and detailed policy has not yet been decided regarding the written policy.

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## NEW TRENDS IN LIBRARY AND INFORMATION SCIENCE RESEARCH IN INDIA

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**Abstract :** *In this article the researcher discusses the new trends in library and information science research in India. It has been analyzed in details. This article covers the study process of knowledge, classification, cataloguing, documentation, library administration, Internet, information literacy, change management, and library security. This shows that the utility and security of libraries is continuously increasing through programmers and mass literacy programs etc. There has been a drastic change in LIS education and training facilities in India and the need to store information and knowledge in libraries.*

*This article is for those currently pursuing BLIS and MLIS as well as Ph.D. Useful for students, research as well as library staff for knowledge.*

**Keywords :** *research, research in LIS, research trends, etc.*

---

### **Introduction :**

Research is searching of innovative idea or principles; intelligent & diligent analysis classification in order to ascertain something. In India, individual research started near about in 1930s with Dr. S.R. Ranganathan, who was actively involved in various areas of library and information science profession; like cataloguing, documentation, library administration, etc. About face is the dominant factor of human life. There is inestimable to change in present period and it is the cause of human progress. The LIS is no exception to this. Therefore, it is automatic to include new trends in the syllabi. As a result of this change new concepts are coming forward. In the age of demolition of information science, the task of fulfilling the reader's expectations totally depend upon the skillful librarian.

To prepare a skilled librarian the library organization and the syllabus it is very important. Now a day's library is not only confined to the transaction of books, but it has become a knowledge resource center. Therefore, it is very needful to apply the new trends in the syllabi.

According to National Knowledge Commission (NKC) report (March 2007), "India has a long tradition of libraries & has contributed to the development of basic concepts in the discipline such as fundamental principles of library services and knowledge organization tools, Research and Development activities in LIS today are extremely limited. This is utmost concern for the growth of Library and information science profession in India"

Research is a very careful critical study or examination in seeking facts or principles, intelligent and diligent investigation in order to ascertain something.

### 1. What is Research?

Research has been defined in a number of different ways. A broad definition of research is given by Martyn Shuttleworth - "In the broadest sense of the word, the definition of research includes any gathering of data, information and facts for the advancement of knowledge."

Another definition of research is given by Creswell who states that - "Research is a process of steps used to collect and analyze information to increase our understanding of a topic or issue".

It consists of three steps: Pose a question, collect data to answer the question, and present an answer to the question.

The Merriam-Webster Online Dictionary defines research in more detail as "a studious inquiry or examination; especially : investigation or experimentation aimed at the discovery and interpretation of facts, revision of accepted theories or laws in the light of new facts, or practical application of such new or revised theories or laws".

Library science research in India started with Dr. S.R. Ranganathan in 1931. So the formal institution of the doctoral degree program in science is unquestionably credited to Dr. S.R. Ranganathan (1892-1972). He formulated five principles of library science. He categorized them as general principles, fundamental laws, theories, principles and rules. Dr. S.R. Ranganathan single-handedly broke new ground in library science and pioneered new experiments. Dr. Ranganathan's tenure is characterized by a period of intellectual contributions to library science, particularly the theory of library classification. First PhD in Library Science Dr. DB Dr. Krishna Rao Awarded in 1957 for a study on 'Facet Analysis and Depth Classification' of Agriculture under the guidance of S.R. Ranganathan & S. Dasgupta. Dr. J.S. Sharma was the first Indian and one of the first few to receive a doctorate from the USA. He was awarded a PhD by the University of Michigan for his thesis 'Mahatma Gandhi: A Descriptive Bibliography'. The second PhD in LIS in India was done in 1977 by Dr. Pandey S.K. was provided to him. Sharma for his thesis 'Extension and Modification of Dewey Decimal Classification for Classifying Indian Books with Special Reference to Indian Philosophy and Indian Religion (18th Edition)' by Punjab University.

### 2. New Trends in LIS Research :

In the past research in library and information science research was mainly considered to provide a theoretical basis for professional practice as research in LIS professions appears to lack deep roots. In the field of library science research, the US premiered Eleanor's Royal Commission on Historical Manuscripts. S. Upton was awarded a Ph.D. in 1930 by the University of Chicago. Research in Library and Information Science is considered the first



doctoral degree in library science. In the 1950s, the universities of Illinois, Michigan, Columbia, Western Reserve, California and Rutgers produced 129 Ph.D.

### **3. Current Trends in LIS :**

#### **3.1. Knowledge of Computer and Internet :**

Today's is the computer generation one who has no knowledge of, computer" is regarded as illiterate. That is why included syllabus for the computer in Library and Information Science. The knowledge of internet is the need of the generation. Because Internet is a most important factor for the current generation. The future Librarian must be given the knowledge of this important technique.

#### **3.2. Use of Computer Cataloguing, database and Information Technology in Syllabus :**

With the use of computer the old topics like classification; cataloguing and database are thought in a new fashion now a day. The information technology functions and manages the information technology. It is also included in the syllabus of Library and Information Science. Its main purpose is to growth and motivates people to know about it.

#### **3.3. Change in Management Science Studies with Library Security :**

The recent trends in management studies, includes the use of laws and knowledge regarding the storage of information and its application. The Librarian has a cautious regarding the library security, they are to be made acquainted with a new techniques. These include for magnetic security system, CCTV and RFID etc.

#### **3.4. Knowledge of Information Production and Sell skills :**

The librarian is expected to bear the skills related to the spell of knowledge production. They must be aware for the fact that, knowledge is never available without charge but some fee must be charged for it.

#### **3.5. Librarian's Training Classes, New Material to the Librarian Fulfilling Reader's Need :**

It is desired that the Librarian ought to be well equipped with communication skills for this, training classes for Librarian are arranged to make him adept in communication skills. The needs of the reader and the information they want can be fulfilled with this. For acquiring information such as internet, website, e-resources is needed to be introduced to the librarians.

#### **3.6. The Library and Information Science Education is distance education mode and Arrangement of Refresher Course :**

The distance education has made it possible for many people to take education while doing a job. Example - IGNOU, New Delhi this are central University. In order to impart the

current information to the Librarians, the University Grand Commission has organized refresher courses for time to time.

**3.7. Arrangement of Continuous education, seminars, workshops and conferences :**

The University and the Library association take initiative in the arrangement of continuous education, seminars, workshops and conferences. The main purpose is to make librarian acquainted with new trends and developments in Library and Information science profession.

**3.8. Information Literacy Programmed and use information and document retrieval:**

It is really very difficult to reach up to the expectations of readers. Therefore, it became the need of the time to arrange information Literacy programmed. It will ultimately provide the librarian with some specific skills. Expert systems Use of automatic classification techniques for document retrieval Application of parallel processing techniques to information retrieval, etc.

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- Ghost writing
- Outsourcing of data processing
- Data cooking Rampant plagiarism
- Cloud libraries
- Mobile applications
- Expert system
- Web interface
- Paper less library
- Library education in digital or web era etc.

**Conclusion :**

At present work of the Library is not confined only to the transaction of the books, but it has become the knowledge resource center of information. Therefore, it has now become mandatory to the follow current trends in the syllabus. With the current trends in the Library and Information Science, the knowledge of computer science has got immense importance ultimately resulting in the need of the use of Internet.



Now days, classification and cataloguing have got immense importance. Curriculum changes have made it necessary to maintain better coordination between information technology and management sciences. New Information production and sell should get a due reference in the syllabus to achieve this goal, the training courses of this sort are run. As a part of this, there is a demand for library and information science education. The education of this sort is also impacted by distance learning mode.

Besides this, it is found that the utility and security of Libraries is steadily increasing by means of the programmers, like continuous education, seminars, conferences and mass Literacy programmed etc.

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# Diamagnetic Al<sup>3+</sup> Doped Ni–Zn Spinel Ferrite: Rietveld Refinement, Elastic, Magnetic, Mössbauer, and Electrical Explorations

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## Abstract

Ni<sub>0.65</sub>Zn<sub>0.35</sub>Al<sub>x</sub>Fe<sub>2-x</sub>O<sub>4</sub> that has been synthesized using a solution-gelation method and calcined at 600 °C for 4 h was characterized using X-ray diffraction (XRD), Fourier transform infrared (FTIR) spectroscopy, scanning electron microscopy (SEM), and transmission electron microscopy (TEM). A single-phase cubic spinel structure belonging to the Fd-3 m space group was confirmed by Rietveld refinement. The cation distribution that was anticipated using the XRD data deviated from the preferential occupancy, and the results of the magnetization analysis supported this. The two absorption bands in the FTIR spectra corresponding to the tetrahedral and octahedral sites further support the establishment of the ferrite skeleton. Al<sup>3+</sup> doping was found to have a considerable impact on the Debye temperature, bulk modulus, and stiffness modulus measured using FTIR data. Interatomic bonding became stronger, increasing elastic moduli. The morphology was examined using SEM, whose results showed a cluster of grains. Additionally, spherical nanoparticles with an average size of 28 nm were visible in the TEM image, which is in good agreement with the crystallite size given by the Williamson-Hall method. The Mössbauer analysis and M–H data showed a soft magnetic behavior with coercivity fluctuation. According to Arrhenius plots, all samples displayed a semiconducting characteristic. With Al<sup>3+</sup> doping, dielectric studies revealed a declining trend.

**Keywords** Rietveld · Cation distribution · Coercivity · Mössbauer · DC Resistivity · Dielectric loss

## 1 Introduction

Metal oxides and ferric oxides have diverse applications [1–5], which make up ferrites, are classified as spinel, hexagonal ferrites and garnet. Transformers, filters, digital

tapes, surface mounting devices, ferroelectric and piezoelectric devices, medical imaging, anti-microbial activity, antennas, information storage devices, inductors, ferrofluids, magneto-caloric refrigeration, battery electrodes, magnetic hyperthermia, etc. are a few examples of these applications [6–13]. For, spinel ferrites, the most notable result is the strong relationship between metallic occupancy over interstitial sites and the method of preparation for structure, morphology, magnetic, Mössbauer, electrical, optical and dielectric properties. Two interstitial sites: tetrahedral (A) and octahedral [B] coordinated with four and six oxygen atoms, respectively, are present in the spinel structure. The typical representations of the overall distribution of cations of the general formula  $MFe_2O_4$  over the (A) and [B] sites are  $(M_{1-x}^{2+}Fe_x^{3+})$  and  $[M_x^{2+}Fe_{2-x}^{3+}]O_4^{2-}$ , respectively. The divalent cation in this instance is designated as "M" and the inversion parameter is "x". They are categorized into three classes based on the inversion parameter: normal, inverse, and mixed when  $x=0$ ,  $x=1$  and  $0 < x < 1$  respectively. It is important to note that the synthesis method and preparation variables have a significant impact on the cation distribution in addition to the atomic size of iron, valences, and

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crystal field. As a result, academicians and scientists are ever improving the preparation processes [14–17] to get these materials with better qualities. These methods of synthesis include co-precipitation, hydrothermal, micro-emulsion, sol–gel, etc. The sol–gel self-combustion method is the most promising of these methods due to its low processing temperature, high yield, high product purity and crystallinity, improved homogeneity, and ultrafine particle production.

Numerous studies have looked into the structural, magnetic, and electrical properties of Ni–Zn nanocrystalline spinel ferrites [18–21] for their potential use in high frequency devices, gas sensing, magneto-dielectric, magneto-optic, magneto-caloric, as RADAR absorbing materials, and other activities [22–24]. Ni–Zn spinel ferrites are naturally ferrimagnetic materials possessing mixed crystal structure belonging to the  $Fd\bar{3}m$  space group. The chemical stability, high permeability, moderate magnetism, high Curie temperature, low coercivity, and low dielectric losses are further characteristics of Ni–Zn spinel ferrites. Numerous studies have shown that the technique of preparation, sintering temperature, type, and concentration of dopant all affect the magnetic properties of Ni–Zn spinel ferrites. Therefore, by changing the preparation process and substituting appropriate metal ions, Ni–Zn ferrites can have their physical, magnetic, Mössbauer, optical, electrical, dielectric, etc. properties modified.

$Ni_{1-x}Zn_xFe_2O_4$  was produced by Deshmukh et al. [25] using a urea-assisted combustion process, and examined its structural, magnetic, and Mössbauer properties. According to experimental findings, a single-phase cubic spinel structure formed. Up to  $x = 0.4$ , magnetization enhanced, and for other values of  $x$ , it decreased. The linewidth widened due to dispersion of the hyperfine field. The Mössbauer finding supports the M–H results. This study revealed that the  $Ni_{0.6}Zn_{0.4}Fe_2O_4$  composition has the potential for enhanced properties and technological applications.  $Ni_{0.6}Zn_{0.4}Fe_{1.5}Al_{0.5}O_4$  was made by Massoudi et al. [26] using the sol–gel method and the impact of annealing on its different properties was assessed. As the annealing temperature increased, the produced nanoparticles grew from the nanoscale to the microscale. The physical parameters were tweaked, and the crystallite size varied in relation to the annealing temperature. It is revealed that a key factor in tailoring the properties of spinel ferrites is the substitution of specific cations. Qing Ni et al. [27] looked into the synergistic effects of Li–Al codoping on the microstructure, magnetic and dielectric properties of Ni–Zn ferrites. The grain development and crystal structure were significantly influenced by the amount of codoping. By using resonant stimulation, Gray et al. [28] showed that the spinel complex  $Ni_{0.65}Zn_{0.35}Fe_{1.2}Al_{0.8}O_4$  is an excellent source of spin current. For the first time, an aluminium-substituted Ni–Zn system was prepared by Dessai et al. [29] utilizing malic acid as a fuel. The single-phase cubic spinel structure development

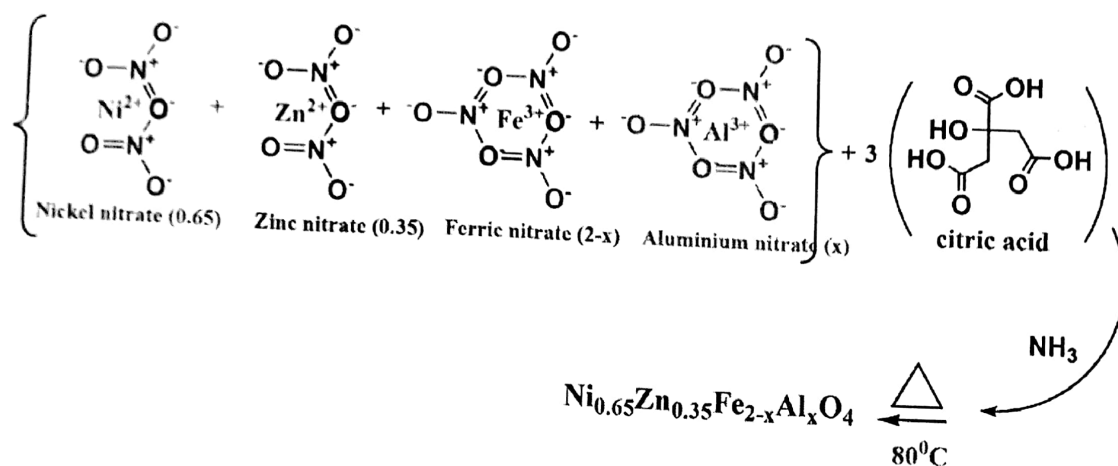
was verified by the structural analysis. All of the components of the prepared spinel ferrites were present as mapped by EDS mapping. It was discovered that the Curie temperature decreased with the concentration of aluminium.

Other metal oxides that have been doped with various dopants and made using various synthesis techniques imply that the physicochemical and other properties have been significantly influenced. These materials are excellent for a variety of technical applications, such as microwave absorption, supercapacitor electrodes, and electromagnetic interference (EMI) shielding etc. [30–34]. Thus, to improve the physical properties of materials and increase their appropriateness for various applications, it is crucial to choose the appropriate substituent [35].

Superexchange interactions, which may be further strengthened by trivalent  $Al^{3+}$  ions, are responsible for the altered magnetic and dielectric properties of Ni–Zn ferrites. It is a well known fact that changing a nonmagnetic material, such  $Fe^{3+}$  to  $Al^{3+}$ , raises resistance and lowers saturation magnetization, changing both the electrical and magnetic properties. All things considered, it can be said that the insertion of  $Al^{3+}$  ions results in a major modification in structure and cationic organization. Furthermore, this helps to lower magnetic coercivity and dielectric loss. In light of this, the present study concentrates on the preparation of nanoparticles and how the trivalent  $Al^{3+}$  ions impact the structure and cationic organization of  $Ni_{0.65}Zn_{0.35}Al_xFe_{2-x}O_4$  with  $x = 0.0, 0.5, \text{ and } 1.0$ . Furthermore, it aims to assess the effects of structure and cation distribution on elastic, magnetic, Mössbauer, DC electrical and dielectric properties via  $Al^{3+}$  inclusion.

## 2 Experimental

Ni–Zn ferrite that has  $Al^{3+}$  ( $Ni_{0.65}Zn_{0.35}Al_xFe_{2-x}O_4$  with  $x = 0.0, 0.5, \text{ and } 1.0$ ) integrated into it was prepared using the sol–gel self-combustion approach. Citric acid ( $C_6H_8O_7$ ), a reducing agent of AR grade, and all nitrates, a source of metal ions, were utilized as received with no further purification. The respective metal nitrates and fuel ratio was chosen in a 1:3 in accordance with a propellant chemistry technique. Separately, stoichiometric amount of  $C_6H_8O_7$  and each metal nitrate was dissolved in distilled water. Then, these various solutions were combined in a beaker and stirred for few minutes. Ammonium solution was added drop by drop to adjust the pH level to a neutral range (7 pH). The wet gel was then created by heating this solution at 80 °C for 4 h. The temperature was further increased to 120 °C once the viscous gel had formed. The viscous gel started to bubble as soon as all of the water molecules evaporate. A little while later, it began to fire and burn alongside the blazing flints. The auto combustion was finished quickly, producing soft ash. Our past reports provide a more thorough explanation of the synthesis process [19, 36]. A schematic of the chemical reaction to be carried out is presented as follows.



A mortar and pestle was used to grind the loose powder. It was then heated at 600 °C for 4 h to improve crystallinity. With the aid of advanced equipment, the annealed powder was characterized for structural, morphological, elastic, magnetic, Mössbauer, DC electrical and dielectric investigations.

## 2.1 Characterization

Using a Cu target, K $\alpha$  radiation, 40 kV, and 40 mA a Philips X-ray diffractometer (XRD), all samples were characterized. The cation distribution and several structural parameters were inferred from the XRD results. A Perkin Elmer spectrometer was used to conduct FTIR analyses of all samples in the 400–4000 cm<sup>-1</sup> range. The elastic properties were assessed using FTIR data. For morphological analyses, a scanning electron microscope (SEM, JEOL JSM 6360) was used. Furthermore, a transmission electron microscope (TEM, Philips, CM 200) was used to determine the morphology and particle size. A pulse field hysteresis tracer was used to measure the magnetization as a function of the applied magnetic field, which was  $\pm 5000$  Oe at room temperature. The Mössbauer measurements were also performed using a <sup>57</sup>Co source and a Mössbauer spectrometer (FAST Com Tec 070906), and each spectra was examined using the MossWinn 4.0 application. A natural iron absorber served as the velocity calibrator. A two-probe technique was used to measure the DC electrical properties as a function of temperature in the range from ambient temperature to 850 K. The electrical parameters such as DC resistivity, activation energy, drift mobility, charge carrier concentration, and diffusion coefficient were obtained using the DC electrical measurements. Furthermore, a two-probe technique was used to measure the dielectric properties as a function of frequency in the range of 50 Hz to 1 MHz. The dielectric constant for

each sample was calculated using capacitance that was measured in parallel.

## 3 Results and Discussion

### 3.1 Rietveld Analysis

The Rietveld refinement patterns of Ni<sub>0.65</sub>Zn<sub>0.35</sub>Al<sub>x</sub>Fe<sub>2-x</sub>O<sub>4</sub> for x=0.0, 0.5, and 1.0 samples are shown in Fig. 1. The lines of black, red, and blue, respectively, denote the lines of experimental intensities, computed intensities, and the difference between them. Since the discrepancy between the experimental and calculated XRD patterns is so minor, that they match quite well. Moreover, the XRD pattern showed no extra peaks, supporting pure phase formation across all samples. All of the XRD patterns with the Miller indices (220), (311), (222), (400), (420), (511), (440), (620), and (533) attest to the creation of a single-phase cubic spinel structure that belongs to the Fd-3 m space group. Additionally, the refinement parameters are also included in each refined XRD pattern. These numbers show that the refinement is in line with the experimental results. The different structural parameters, including the lattice parameter (a), crystallite size (D), X-ray density (d<sub>x</sub>), porosity (P), dislocation density (δ), and strain (ε) of all samples, were calculated from the XRD data using formulas given in [37]. These results are listed in Table 1.

$$a = d\sqrt{h^2 + k^2 + l^2} \text{ \AA} \quad (1)$$

$$D = \frac{0.9\lambda}{\beta \cos\theta} \text{ nm} \quad (2)$$

$$d_x = \frac{Z \times M}{V \times N_A} \frac{\text{gm}}{\text{cm}^3} \quad (3)$$



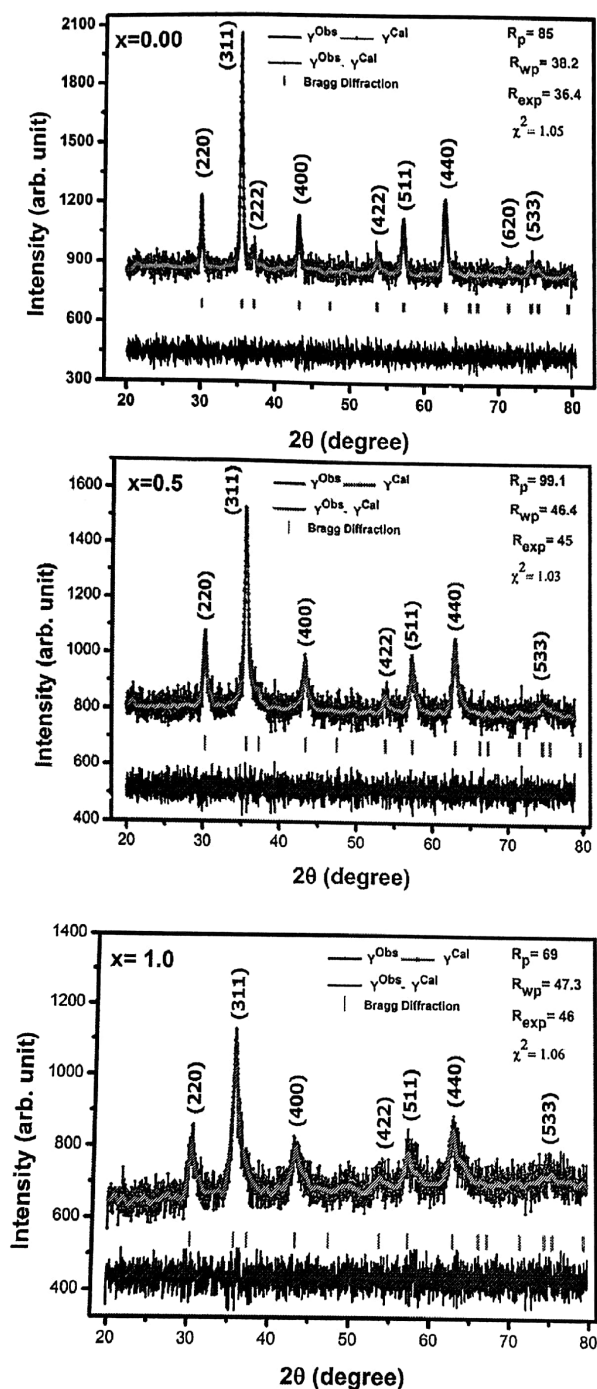


Fig. 1 Rietveld refined XRD patterns of  $\text{Ni}_{0.65}\text{Zn}_{0.35}\text{Al}_x\text{Fe}_{2-x}\text{O}_4$ ,  $x=0.0, 0.5$ , and  $1.0$  samples

Table 1 Structural parameters of  $\text{Ni}_{0.65}\text{Zn}_{0.35}\text{Al}_x\text{Fe}_{2-x}\text{O}_4$  ( $x=0.0, 0.5$  and  $1.0$ ) samples

| Sample code | a (Å) | V (Å) <sup>3</sup> | d <sub>x</sub> (g/cm <sup>3</sup> ) | d <sub>B</sub> (g/cm <sup>3</sup> ) | P (%) | D (nm) |     | δ (lines/m <sup>2</sup> ) | ε (%)                 |
|-------------|-------|--------------------|-------------------------------------|-------------------------------------|-------|--------|-----|---------------------------|-----------------------|
|             |       |                    |                                     |                                     |       | XRD    | W-H |                           |                       |
| x=0.0       | 8.372 | 586.9              | 5.358                               | 3.638                               | 32    | 25     | 28  | $4.41 \times 10^{15}$     | $1.61 \times 10^{-3}$ |
| x=0.5       | 8.325 | 577.1              | 5.759                               | 3.617                               | 40    | 14     | 17  | $11.45 \times 10^{15}$    | $2.30 \times 10^{-3}$ |
| x=1.0       | 8.298 | 571.6              | 6.129                               | 3.587                               | 46    | 10     | 15  | $38.71 \times 10^{15}$    | $7.10 \times 10^{-3}$ |

$$P = 1 - \frac{d_B}{d_X} \% \quad (4)$$

$$\delta = \frac{1}{l^2} \text{lines/m}^2 \quad (5)$$

$$\varepsilon = \frac{\beta \cos \theta}{4} \quad (6)$$

where,  $d$  interplanar spacing,  $(h, k, l)$  Miller indices,  $\lambda$  wavelength,  $\beta$  full width at half maxima,  $\theta$  angle,  $Z$  number of atoms per unit cell,  $M$  molecular weight,  $N_A$  Avogadro number,  $d_B$  bulk density and  $t$ -average particle size

The lattice parameter decreased with the  $\text{Al}^{3+}$  ion content. The observed decrease was attributed to the ionic radii of  $\text{Fe}^{3+}$  and  $\text{Al}^{3+}$  ions being different. The decrement is caused by the substitution of lower ionic radii  $\text{Al}^{3+}$  ions (0.064 nm) for higher ionic radii  $\text{Fe}^{3+}$  ions (0.067 nm). This decrease is consistent with studies in the literature [38]. Scherrer's formula and Williamson-Hall (W-H) plot (Fig. 2) study showed that the average crystallite size reduced with an increase in  $\text{Al}^{3+}$  ions, from 25 to 10 nm and 28 nm to 15 nm, respectively. The crystallite size values discovered using W-H analysis is in agreement with those discovered through the Debye-Scherrer formula. Table 1 demonstrates that the X-ray density rises as the  $\text{Al}^{3+}$  amount does. The lattice parameter and the X-ray density are inversely connected. Therefore, the drop in lattice parameter can be used to explain the observed rise in X-ray density. Using the Archimedes principle, the bulk densities of all samples were calculated, and the results are shown in Table 1. According to Table 1, bulk density values ranged from 3.638 gm/cm<sup>3</sup> to 3.587 gm/cm<sup>3</sup>. Additionally, it is noted that the bulk density values are significantly lower than the X-ray density values, leading to high porosity values. Because of the increasing value of the X-ray density, the % porosity computed from relation (5) reported in Table 1 enhanced with the  $\text{Al}^{3+}$  content. Furthermore, the agglomeration of particles during the synthesis is to account for the high values of porosity (32% to 46%). According to relation (6), the dislocation density ( $\delta$ ) values of all samples fall between  $4.41 \times 10^{15}$  and  $38.71 \times 10^{15}$  lines/m<sup>2</sup>. Table 1 lists the lattice strain values, which range from  $1.61 \times 10^{-3}$  to  $7.1 \times 10^{-3}$ .

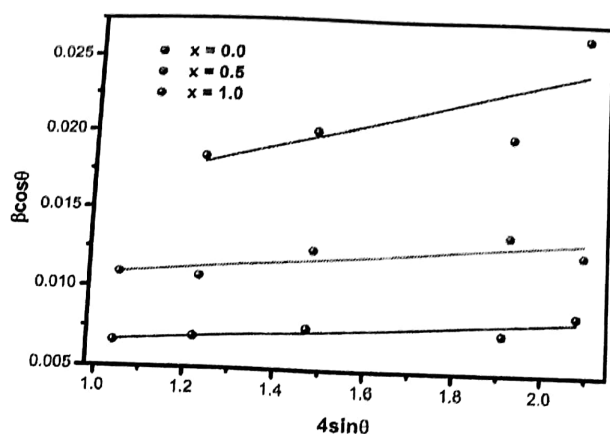


Fig. 2 W-H plots of  $\text{Ni}_{0.65}\text{Zn}_{0.35}\text{Al}_x\text{Fe}_{2-x}\text{O}_4$ ,  $x=0.0, 0.5$ , and  $1.0$  samples

### 3.2 Hopping Length ( $L_A$ and $L_B$ )

The following relations were used to determine the hopping length for the tetrahedral (A) ( $L_A$ ) and octahedral [B] sites ( $L_B$ ) [39].

$$L_A = a\sqrt{3/4} \quad (7)$$

$$L_B = a\sqrt{2/4} \quad (8)$$

Table 2 lists the values for the  $L_A$  and  $L_B$  hopping lengths. Both  $L_A$  and  $L_B$  decrease with  $\text{Al}^{3+}$  content, as shown in Table 2. The reduction in the lattice parameter with  $\text{Al}^{3+}$  content is the cause of the decrease in hopping length.

The standard relations provided were used to calculate the tetrahedral bond length ( $d_{AX}$ ), octahedral bond length ( $d_{BX}$ ),

tetra edge ( $d_{AXE}$ ), octa edge ( $d_{BXE}$ ), and ( $d_{BEU}$ ) for all samples  $d_{AX} = a\sqrt{3(u - \frac{1}{4})}$ ,  $d_{BX} = a\sqrt{3u^2 - \frac{11}{4}u + \frac{43}{64}}$ ,  $d_{AXE} = a\sqrt{2(2u - \frac{1}{2})}$ ,  $d_{BXE} = a\sqrt{2(1 - 2u)}$ ,  $d_{BEU} = a\sqrt{4u^2 - 3u + \frac{11}{16}}$ .

Table 2 provides the values for each of these structural parameters. All of these metrics decrease with increasing  $\text{Al}^{3+}$  content, as seen in Table 2. This is so because the lattice parameter directly affects these parameters.

### 3.3 Ionic Radii ( $r_A$ and $r_B$ )

The lattice parameter "a" and the oxygen positional parameter "u" (0.381) can be used to compute the ionic radius of the tetrahedral A-site ( $r_A$ ) and the octahedral B-site ( $r_B$ ), respectively. Table 2 lists the tetrahedral and octahedral ionic radii's respective values.

$$r_A = \left(u - \frac{1}{4}\right)a\sqrt{3} - r(\text{O}^{2-}) \quad (9)$$

$$r_B = \left(\frac{5}{8} - u\right)a - r(\text{O}^{2-}) \quad (10)$$

where the radius of oxygen anions is shown by the symbol  $r(\text{O}^{2-})$ . Table 2 lists the tetrahedral and octahedral ionic radii values. Both of these values fall as the  $\text{Al}^{3+}$  content rise.

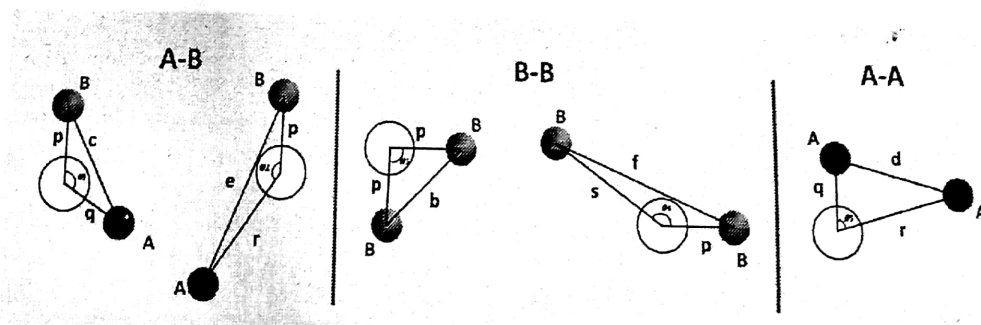
### 3.4 Interionic Distances and Bond Angles

The spinel ferrite structure is presented schematically in Fig. 3 together with a bond length and bond angle [40]. The interionic distance between ions, i.e. the cation-cation (Me-Me) distances (b, c, d, e, f) and cation-anion (Me-O) distances (p, q, r, s), were computed taking into account the experimentally observed values of the lattice parameter and

Table 2 Structural parameters of  $\text{Ni}_{0.65}\text{Zn}_{0.35}\text{Al}_x\text{Fe}_{2-x}\text{O}_4$  ( $x=0.0, 0.5$  and  $1.0$ ) samples

| Sample code | $L_A$ (Å) | $L_B$ (Å) | $d_{AX}$ (Å) | $d_{BX}$ (Å) | $d_{AXE}$ (Å) | $d_{BXE}$ (Å) | $d_{BEU}$ (Å) | $r_A$ (Å) | $r_B$ (Å) |
|-------------|-----------|-----------|--------------|--------------|---------------|---------------|---------------|-----------|-----------|
| $x=0.0$     | 3.625     | 2.960     | 1.899        | 2.044        | 3.102         | 2.818         | 2.961         | 0.579     | 0.723     |
| $x=0.5$     | 3.605     | 2.435     | 1.889        | 2.032        | 3.084         | 2.802         | 2.945         | 0.569     | 0.711     |
| $x=1.0$     | 3.593     | 2.428     | 1.883        | 2.026        | 3.075         | 2.793         | 2.936         | 0.563     | 0.704     |

Fig. 3 Schematic for interionic distances and bond angles of  $\text{Ni}_{0.65}\text{Zn}_{0.35}\text{Al}_x\text{Fe}_{2-x}\text{O}_4$ , ( $x=0.0$ )





**Table 3** Interionic distance values (p, q, r, s, b, c, d, e and f) of  $\text{Ni}_{0.65}\text{Zn}_{0.35}\text{Al}_x\text{Fe}_{2-x}\text{O}_4$  ( $x = 0.0, 0.5$  and  $1.0$ ) samples

| Sample code | p (Å) | q (Å) | r (Å) | s (Å) | b (Å) | c (Å) | d (Å) | e (Å) | f (Å) |
|-------------|-------|-------|-------|-------|-------|-------|-------|-------|-------|
| x = 0.0     | 2.043 | 1.898 | 3.636 | 3.654 | 2.961 | 3.471 | 3.625 | 5.438 | 5.127 |
| x = 0.5     | 2.031 | 1.888 | 3.617 | 3.633 | 2.943 | 3.451 | 3.605 | 5.407 | 5.098 |
| x = 1.0     | 2.024 | 1.883 | 3.605 | 3.622 | 2.934 | 3.441 | 3.593 | 5.391 | 5.082 |

oxygen positional parameter ( $u = 0.381$  Å). These interionic distances are crucial in determining the magnetic properties and are helpful in describing the crystallographic structure. The following relations are used to estimate the interionic bond distances between cations and cation-anions, and their values are provided in Table 3:

### 3.5 Me–Me distances

$$b = \sqrt{2} \left( \frac{a}{4} \right)$$

$$c = \sqrt{11} \left( \frac{a}{8} \right)$$

$$d = \sqrt{3} \left( \frac{a}{4} \right)$$

$$e = \sqrt{3} \left( \frac{3a}{8} \right)$$

$$f = \sqrt{6} \left( \frac{a}{4} \right)$$

### 3.6 Me–O distances

$$p = a \left( \frac{5}{8} - u \right)$$

$$q = a \sqrt{3} \left( u - \frac{1}{4} \right)$$

$$r = a \sqrt{11} \left( u - \frac{1}{4} \right)$$

$$s = a \sqrt{3} \left( \frac{u}{3} + \frac{1}{8} \right)$$

According to Table 3, the interionic distances all shorten as the  $\text{Al}^{3+}$  content rises. The interionic connection should become stronger when the Me–Me and Me–O distances are reduced. The literature [41] contains similar findings for sol–gel combustion-produced nanocrystalline spinel ferrites. The following relations are used to determine the bond angles, namely  $\theta_1, \theta_2, \theta_3, \theta_4$  and  $\theta_5$ , between the cations and cation-anions:

#### 3.6.1 Bond angles

$$\theta_1 = \text{Cos}^{-1} \left( \frac{p^2 + q^2 - c^2}{2pq} \right)$$

$$\theta_2 = \text{Cos}^{-1} \left( \frac{p^2 + r^2 - e^2}{2pr} \right)$$

$$\theta_3 = \text{Cos}^{-1} \left( \frac{2p^2 - b^2}{2p^2} \right)$$

$$\theta_4 = \text{Cos}^{-1} \left( \frac{p^2 + s^2 - f^2}{2ps} \right)$$

$$\theta_5 = \text{Cos}^{-1} \left( \frac{r^2 + q^2 - d^2}{2rq} \right)$$

The calculated bond angles are shown in Table 4, which indicates that  $\theta_1, \theta_2$ , and  $\theta_5$  decrease with  $\text{Al}^{3+}$  content, while bond angles  $\theta_3$  and  $\theta_4$  increase with  $\text{Al}^{3+}$  substitution. This behavior can be attributed to the A–A and A–B exchange interaction being weaker, while the B–B exchange interaction becoming stronger. The variations in bond angle values

**Table 4** Bond angle values ( $\theta_1, \theta_2, \theta_3, \theta_4, \theta_5$ ) of  $\text{Ni}_{0.65}\text{Zn}_{0.35}\text{Al}_x\text{Fe}_{2-x}\text{O}_4$  ( $x = 0.0, 0.5$  and  $1.0$ ) samples

| Sample code | $\theta_1$ (degrees) | $\theta_2$ (degrees) | $\theta_3$ (degrees) | $\theta_4$ (degrees) | $\theta_5$ (degrees) |
|-------------|----------------------|----------------------|----------------------|----------------------|----------------------|
| x = 0.0     | 123.355              | 145.017              | 92.828               | 125.912              | 74.523               |
| x = 0.5     | 123.343              | 144.961              | 92.848               | 125.916              | 74.489               |
| x = 1.0     | 123.340              | 144.946              | 92.853               | 125.918              | 74.481               |

are attributed to cation rearrangement with varying superexchange interactions. As a result, we can anticipate a decrease in magnetization with  $Al^{3+}$  replacement owing to changes in bond angles.

### 3.7 Cation Distribution

The cation distribution was established to establish the site occupancy of the cations over interstitial sites (A) and [B], which is highly beneficial for understanding the structural features of spinel ferrites. For this objective, the relative intensity calculations approach employing Eq. 11 [42] was used to calculate the intensity ratios for all samples.

$$I_{hkl} = |F|_{hkl}^2 P L_p \quad (11)$$

where  $I_{hkl}$  is the intensity ratio,  $F$  is the structure factor,  $P$  is the multiplicity factor and  $L_p$  is the Lorentz-polarization factor. The multiplicity factor  $P$  for powder photographs of the cubic system, i.e., for 422, 440, 220 and 400 plane multiplicities of 24, 12, 12 and 6 Å, respectively. Only Bragg's diffraction angle determines Lorentz polarisation, which is determined by Eq. 12 [43].

$$L_p = \left[ \frac{1 + \cos^2 2\theta}{\sin^2 \theta \cos \theta} \right] \quad (12)$$

For the intensity ratio calculations, the (hkl) planes (220), (400), and (422) were taken into account.  $Ni_{0.65}Zn_{0.35}Fe_{2-x}Al_xO_4$  ( $x=0.0, 0.5, \text{ and } 1.0$ ) samples cation distribution were estimated using some of the peak intensity ratio combinations with the highest sensitivity for cation distribution, such as  $I_{400}/I_{220}$ ,  $I_{422}/I_{220}$ , and  $I_{422}/I_{400}$  [44, 45]. These intensity ratio combinations and the measured intensity ratios were compared. Although there is some discrepancy between the estimated and observed intensity ratios

(Table 5), the closer agreement between ratios was deemed to represent the ideal cation distribution, and Table 6 reflects this.

Table 6 demonstrates that on the  $Al^{3+}$  ions incorporation, nickel ions occupied both the (A) and [B] sites whereas the divalent zinc ions heavily moved to the [B] locations. Previous investigations [46–49] have also noted this occupancy preference for spinel ferrites synthesized via the sol-gel auto combustion method, particularly for  $Zn^{2+}$  ions. Additionally, Table 6 demonstrates that  $Al^{3+}$  is present at both the (A) and [B] sites. Ni–Zn ferrites are reported to have a mixed spinel structure, with  $Zn^{2+}$  having strong preference for tetrahedral (A) sites,  $Ni^{2+}$  and  $Fe^{3+}$  having preference for both (A) and (B) sites, and  $Al^{3+}$  having preference for (B) sites. The surface energy is insufficient at a nanoscale dimension to locate the cations in their typical locations. Redistribution of cations in contrast to their typical occupancy is thus feasible. This results in extraordinary properties due to the size-dependent cation dispersion. Additionally, the estimated cation distribution was used to calculate the magneton number ( $B_{cal}$ ) for all three samples, which matches the observed magneton number ( $B_{obs}$ ) calculated using magnetization data quite well. As a result, both the estimated cation distribution from magnetization data and that from XRD data are validated and reported in Table 6.

### 3.8 FTIR

The successful development of spinel structures and the existence of the other functional entities in  $Al^{3+}$ -substituted Ni–Zn spinel ferrites were investigated using Fourier transform infrared (FTIR) spectroscopy. The transmittance spectra captured in the wavenumber range of  $350\text{ cm}^{-1}$  to  $4000\text{ cm}^{-1}$  are shown in Fig. 4. The spectra contain the two dominant vibrational mode frequencies ( $\nu_1$

**Table 5** Intensity ratios and their differences used for the estimation of cation distribution of  $Ni_{0.65}Zn_{0.35}Al_xFe_{2-x}O_4$  ( $x=0.0, 0.5$  and  $1.0$ ) samples

| Sample  | $I_{400}/I_{220}$ |        |        | $I_{422}/I_{220}$ |        |        | $I_{422}/I_{400}$ |        |        |
|---------|-------------------|--------|--------|-------------------|--------|--------|-------------------|--------|--------|
|         | obs               | cal    | diff   | obs               | cal    | diff   | obs               | cal    | diff   |
| $x=0.0$ | 0.9017            | 0.9006 | 0.0011 | 0.7981            | 0.4679 | 0.3302 | 0.8862            | 0.5188 | 0.3673 |
| $x=0.5$ | 0.9135            | 0.8623 | 0.0512 | 0.8045            | 0.4703 | 0.3342 | 0.8807            | 0.5454 | 0.3353 |
| $x=1.0$ | 0.9958            | 0.9586 | 0.0372 | 0.8692            | 0.4776 | 0.3916 | 0.9067            | 0.4653 | 0.4272 |

**Table 6** Cation distribution from XRD and magnetization as well as magneton number (experimental (Obs) and theoretical (Cal)) of  $Ni_{0.65}Zn_{0.35}Al_xFe_{2-x}O_4$  ( $x=0.0, 0.5$  and  $1.0$ ) samples

| Sample  | Cation distribution from XRD             |  | Cation distribution from Magnetization   |  | $\eta_B$ ( $\mu_B$ )              |      |
|---------|--|--|--|--|-----------------------------------|------|
|         | (A) site                                 | [B] site                                 | (A) site                                 | [B] site                                 | Obs                               | Cal  |
|         | $x=0.0$                                  | $(Ni_{0.14}Zn_{0.03}Fe_{0.83})$          | $[Ni_{0.51}Zn_{0.32}Fe_{1.17}]$          | $(Ni_{0.15}Zn_{0.024}Fe_{0.826})$        | $[Ni_{0.50}Zn_{0.326}Fe_{1.174}]$ | 2.45 |
| $x=0.5$ | $(Ni_{0.11}Zn_{0.09}Al_{0.11}Fe_{0.68})$ | $[Ni_{0.53}Zn_{0.26}Al_{0.39}Fe_{0.82}]$ | $(Ni_{0.11}Zn_{0.09}Al_{0.11}Fe_{0.68})$ | $[Ni_{0.53}Zn_{0.26}Al_{0.39}Fe_{0.82}]$ | 1.53                              | 1.52 |
| $x=1.0$ | $(Ni_{0.07}Zn_{0.12}Al_{0.30}Fe_{0.51})$ | $[Ni_{0.58}Zn_{0.23}Al_{0.70}Fe_{0.49}]$ | $(Ni_{0.07}Zn_{0.13}Al_{0.29}Fe_{0.51})$ | $[Ni_{0.58}Zn_{0.22}Al_{0.71}Fe_{0.49}]$ | 0.93                              | 0.92 |



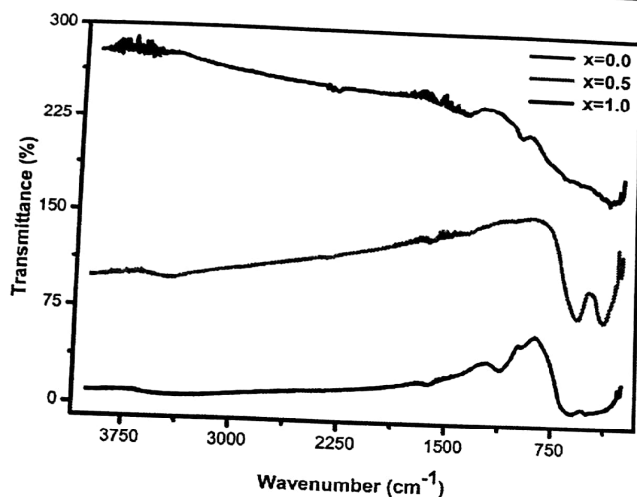


Fig. 4 FTIR spectra of  $\text{Ni}_{0.65}\text{Zn}_{0.35}\text{Al}_x\text{Fe}_{2-x}\text{O}_4$ ,  $x=0.0, 0.5, \text{ and } 1.0$  samples

and  $\nu_2$ ), which are located between 570 and 600  $\text{cm}^{-1}$  and 380 and 400  $\text{cm}^{-1}$ , respectively. Table 7 lists the values of the vibrational mode frequencies  $\nu_1$  and  $\nu_2$ . The stretching at the interstitial tetrahedral (A) and octahedral (B) sublattices is responsible for these vibrational mode frequencies, respectively. Table 7 demonstrates how  $\text{Al}^{3+}$  substitution caused a compositional shift in the vibrational mode frequencies, moving them toward higher levels. This change in frequency amplifies the fact that the generated mixed Ni–Zn spinel ferrite matrix underwent cationic redistribution following the substitution of  $\text{Al}^{3+}$  ions. The accommodation of the  $\text{Al}^{3+}$  ions at both interstitial sublattice

locations is thus shown by the current outcome of shifting in vibrational mode frequencies. The cation distribution calculated from the XRD and magnetization data shows the same thing (Table 6). Additionally, the change in the  $\text{Fe}^{3+}\text{-O}^{2-}$  distance between the octahedral (1.99 Å) and tetrahedral (1.89 Å) complexes is likely to account for the variation in the vibrational band locations [50]. Additionally, using the following equations and the Waldron technique [44], the force constants per atom for the tetrahedral (A) and octahedral (B) sites were calculated:

$$k_t = 7.62 \times M_A \times \nu_1^2 \times 10^{-7} \text{ N/m} \quad (13)$$

$$k_o = 10.62 \times \frac{M_B}{2} \times \nu_2^2 \times 10^{-7} \text{ N/m} \quad (14)$$

where,  $M_A$  and  $M_B$  stand for the average molecular weights of the cations at the (A) and [B] sites respectively. The molecular weights of the (A) and [B]-sites were calculated taking into account the estimated site occupancy and the results are shown in Table 7. Furthermore, Table 7 lists the values for all samples and the Debye temperature (D) determined by Eq. 15.

$$\theta_D = \frac{\hbar c \nu_{mean}}{k} \quad (15)$$

where  $\hbar = h/2\pi$  here  $h$  is Planck's constant,  $c$  is the velocity of light,  $\nu_{mean} = \frac{\nu_1 + \nu_2}{2}$  is the average frequency of principle band positions at the (A) and [B] sites, and  $k$  is the average force constant; its values are given in Table 7. The band positions also move towards higher values as the Debye temperature with  $\text{Al}^{3+}$  content rises proportionally to them.

**Table 7** IR absorption bands  $\nu_1$  and  $\nu_2$ , force constant ( $k_t$ ,  $k_o$ ) at the (A) and [B]-sites, average force constant ( $k$ ) and Debye temperature for  $\text{Ni}_{0.65}\text{Zn}_{0.35}\text{Al}_x\text{Fe}_{2-x}\text{O}_4$  ( $x=0.0, 0.5$  and  $1.0$ ) samples

| Sample  | $\nu_1$<br>( $\text{cm}^{-1}$ ) | $\nu_2$<br>( $\text{cm}^{-1}$ ) | $k_t$<br>(N/m) | $k_o$<br>(N/m) | $k$<br>(N/m) | $\theta_D$<br>(K) |
|---------|---------------------------------|---------------------------------|----------------|----------------|--------------|-------------------|
| $x=0.0$ | 545.00                          | 356.00                          | 127.69         | 78.27          | 102.98       | 648               |
| $x=0.5$ | 572.85                          | 391.54                          | 133.04         | 85.55          | 109.29       | 694               |
| $x=1.0$ | 605.29                          | 413.00                          | 138.58         | 85.34          | 111.96       | 733               |

**Table 8** The bulk modulus (B), longitudinal ( $V_L$ ) transverse ( $V_t$ ) and mean ( $V_m$ ) wave velocities, rigidity modulus (G), and Poisson's ratio (P) for  $\text{Ni}_{0.65}\text{Zn}_{0.35}\text{Al}_x\text{Fe}_{2-x}\text{O}_4$  ( $x=0.0, 0.5$  and  $1.0$ ) samples

| Sample  | B<br>(m/s) | $V_L$<br>(m/s) | $V_t$<br>(m/s) | $V_m$<br>(m/s) | G<br>(GPa) | P<br>(GPa) |
|---------|------------|----------------|----------------|----------------|------------|------------|
| $x=0.0$ | 123.00     | 4791           | 2766           | 3579           | 41.00      | 0.45       |
| $x=0.5$ | 131.44     | 4777           | 2758           | 3569           | 43.81      | 0.48       |
| $x=1.0$ | 134.86     | 4691           | 2708           | 3504           | 44.95      | 0.49       |

### 3.9 Elastic Properties

According to stiffness constants  $C_{11}$  and  $C_{12}$ , the bulk modulus ( $B$ ) is determined as  $B = \frac{1}{3}(C_{11} + 2C_{12})$ . According to Waldron [51], for isotropic materials with cubic structure  $C_{11} \approx C_{12}$ , it may be figured out that  $C_{11} = k/a$ , in this case because  $k$  is the average force constant; therefore  $B = k/a$ . Table 8 lists the values of the bulk modulus. According to Table 8, the bulk modulus increases as the  $\text{Al}^{3+}$  content increases. The calculated values for the longitudinal and transverse elastic wave velocities ( $V_l = \sqrt{C_{11}/d_x}$ ) and ( $V_t = V_l/\sqrt{3}$ ) are shown in Table 8. We discovered that the  $\text{Al}^{3+}$  content causes a drop in both elastic velocities. Additionally, elastic moduli including the rigidity modulus ( $G$ ), Poisson's ratio ( $P$ ), and mean elastic wave velocity ( $V_m$ ) for all samples were computed using the formulas listed below [52], and their values are listed in Table 8

$$V_m = \frac{1}{3} \left( \frac{2}{V_l^3} + \frac{1}{V_t^3} \right)^{-\frac{1}{3}} \quad (16)$$

$$G = d_x V_t^2 \quad (17)$$

$$P = 3B - \frac{2G}{6B} + 2G \quad (18)$$

With more  $\text{Al}^{3+}$  substitution, the elastic modulus values marginally rise, which can be attributed to stronger interatomic bonding between various atoms of the spinel lattice. With  $\text{Al}^{3+}$  substitution, the interatomic bonds between different atoms get stronger over time, increasing the elastic modulus.

### 3.10 SEM and TEM

Figure 5, which shows the porous and foam-like structure, was used to conduct the morphological examinations on all of the samples. Additionally, it can be seen that grains are aggregated. Because of the high surface energy and magnetic interactions between the nanoparticles, aggregations have been formed. A non-uniform grain size can therefore be seen in the SEM images. Moreover, TEM of a typical sample  $\text{Ni}_{0.65}\text{Zn}_{0.35}\text{Fe}_2\text{O}_4$  ( $x=0.0$ ) was carried out, and the TEM image and particle size distribution histogram are shown in Fig. 6. The TEM image amply demonstrates the spherical shape and little agglomeration of the particles. The average particle size as obtained by TEM as presented in TEM histogram is  $\sim 29$  nm, which is consistent with the size of the crystallites as established by XRD data and W-H plots.



Fig. 5 SEM images of  $\text{Ni}_{0.65}\text{Zn}_{0.35}\text{Al}_x\text{Fe}_{2-x}\text{O}_4$ ,  $x=0.0, 0.5,$  and  $1.0$  samples

### 3.11 Magnetization

The M-H hysteresis graphs of each sample taken at room temperature are displayed in Fig. 7. A symmetric hysteresis curve with soft magnetic behavior can be seen in all of the



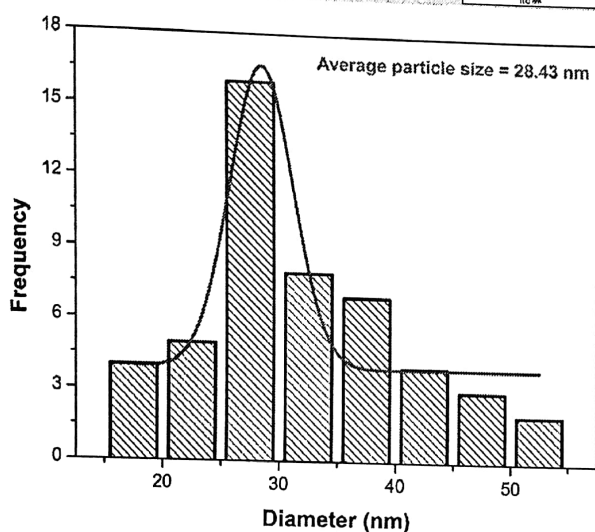
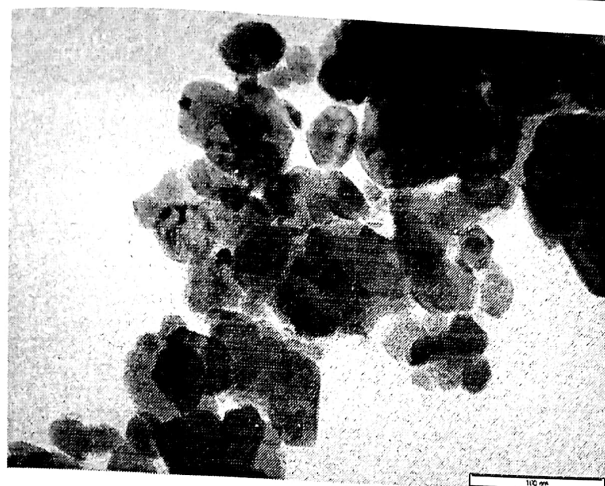


Fig. 6 TEM image and particle size distribution histogram of a typical sample  $\text{Ni}_{0.65}\text{Zn}_{0.35}\text{Fe}_2\text{O}_4$  ( $x=0.0$ )

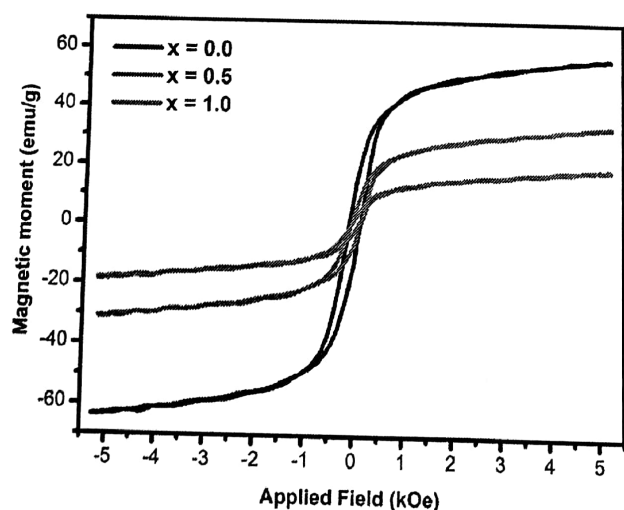


Fig. 7 M-H plots of  $\text{Ni}_{0.65}\text{Zn}_{0.35}\text{Al}_x\text{Fe}_{2-x}\text{O}_4$ ,  $x=0.0, 0.5,$  and  $1.0$  samples

M-H plots. Table 9 lists the values for magnetic properties such as magneton number ( $\eta_B$ ), coercivity ( $H_C$ ), saturation magnetization ( $M_s$ ), and remanence magnetization ( $M_r$ ).  $M_s$  significantly drops with  $\text{Al}^{3+}$  ion substitution, as shown in Table 9. Along with  $M_s$ ,  $M_r$  and  $H_C$  significantly dropped when  $\text{Al}^{3+}$  ions were substituted. The alterations in the (A)-[B] exchange interactions are associated with the decline in these magnetic parameters [29, 53, 54].

There are three exchange interactions, namely (A)-(A), [B]-[B], and (A)-[B], according to Neel's two sublattice ferri-magnetism model, but the (A)-[B] exchange interaction is the dominant one [40]. Although they are not equal in size, the magnetic moments at the (A) and [B] locations line up in an anti-parallel way. The difference between the magnetic moments of the two sublattices, (A) and [B], is therefore the net magnetic moment. The amount of dopant cations and its magnetic moment play a major role in the exchange interaction.  $\text{Fe}^{3+}$ ,  $\text{Al}^{3+}$ ,  $\text{Ni}^{2+}$ , and  $\text{Zn}^{2+}$  have magnetic moments of  $5 \mu_B$ ,  $0 \mu_B$ ,  $2 \mu_B$ , and  $0 \mu_B$ , respectively. Octahedral [B] sites are more frequently occupied by  $\text{Al}^{3+}$  ions. As a result, the (A)-[B] exchange interaction is weakened and the [B]-[B] exchange interaction is expanded when the highly magnetic  $\text{Fe}^{3+}$  ions that were previously present in the octahedral sites are replaced by low magnetic  $\text{Al}^{3+}$  ions. The observed magnetization behavior can be explained using Neel's model  $\eta_B^N = M_B - M_A$  where  $M_B$  and  $M_A$  are magnetic moments at octahedral [B] and tetrahedral (A) sites, respectively.  $\text{Zn}^{2+}$  and  $\text{Ni}^{2+}$  partially filled both (A) and [B] sites, according to the cation distribution inferred from magnetization data (Table 6). Therefore, a divergence from the usual cation preference is seen in the current work. Additionally,  $\text{Al}^{3+}$  substitution at (A) sites took the place of  $\text{Fe}^{3+}$  ions, lowering the magnetic moment there because it has zero magnetic moment. Overall, the saturation magnetization was decreased with  $\text{Al}^{3+}$  because lower magnetic moment  $\text{Al}^{3+}$  ions replaced higher magnetic moment  $\text{Fe}^{3+}$  ions.  $\eta_B = \frac{\text{MolWt} \cdot M_s}{5585}$  yields an experimental magneton number. The values of the theoretical and experimental magneton numbers (Table 6) are in good agreement with one another. The values of remanence and coercivity with  $\text{Al}^{3+}$  substitution in Table 9 only slightly vary, demonstrating the synthesized spinel ferrites soft magnetic properties. The range of the remanence ratio ( $M_r/M_s$ ) was discovered to be between 0.181 and 0.121. Lower values of  $M_r/M_s$  recommended these materials for high frequency devices since they indicated the multidomain character of the particles that may interact through magnetostatic interactions. The magnetic spin orientation is determined by the magnetocrystalline anisotropy ( $H_k$ ), which is calculated using Eq. 19 and binds magnetization in a preferred direction.

**Table 9** Magnetic parameters of  $\text{Ni}_{0.65}\text{Zn}_{0.35}\text{Al}_x\text{Fe}_{2-x}\text{O}_4$  ( $x = 0.0, 0.5$  and  $1.0$ ) samples

| Sample code | $M_s$ (emu/g) | $M_r$ (emu/g) | $H_c$ (Oe) | $M_r/M_s$ | $K$ (Oe) | $H_K$ (erg/cm <sup>3</sup> ) |
|-------------|---------------|---------------|------------|-----------|----------|------------------------------|
| $x = 0.0$   | 57.64         | 10.45         | 109.34     | 0.181     | 227.79   | 6564.95                      |
| $x = 0.5$   | 34.05         | 4.21          | 82.31      | 0.123     | 171.47   | 2918.46                      |
| $x = 1.0$   | 19.58         | 2.37          | 25.74      | 0.121     | 53.63    | 525.01                       |

$$H_K = \frac{2K}{M_s} \quad (19)$$

where  $K$  is the anisotropy constant and  $M_s$  is the saturation magnetization. The saturation magnetization and coercivity are connected by the anisotropy constant. Table 9 displays how magnetocrystalline anisotropy declines with  $\text{Al}^{3+}$  substitution.

### 3.12 Mössbauer Analysis

A strong tool for investigating data on the valence state and magnetic behavior is the Mössbauer technique. It also recognizes where the Fe ions are situated in the nanoferrite system within the crystal structure. In Fig. 8, employing DIST formation, the fitted Mössbauer spectra of all the samples collected at room temperature are shown. They were fitted with one magnetic sextet and one doublet referring to two interstitial sites, tetrahedral (A) and octahedral [B] sites. Table 10 lists the hyperfine parameters derived from fitted Mössbauer spectra. The increase in line width can be attributed to the hyperfine field distribution brought on by the distribution of cations over sublattices. In essence, the isomer shift values tell us something about the oxidation state of Fe-ions. The  $\text{Fe}^{3+}$  valence states are present and the  $\text{Fe}^{2+}$  valence state is denied when the isomer shift values are less than 0.5 mm/s. When compared to tetrahedral sites, octahedral sites have higher isomer shift values. This outcome can be explained by the fact that  $\text{Fe}^{3+}-\text{O}^{2-}$  compared to tetrahedral sites have a higher interionic distance.  $\text{Fe}^{3+}$  ions at octahedral sites are smaller than those at tetrahedral sites as a result of this overlap. As a result, octahedral sites have isomer shift values that are higher than tetrahedral sites. Sextets have incredibly low quadrupole splitting values (Table 10). This is explained by the continued cubic symmetry even after significant  $\text{Al}^{3+}$  ion substitution. Higher values of quadrupole splitting are a result of the noncubic symmetry brought on by the presence of  $\text{Fe}^{2+}$  ions. The magnetic moment, which with  $\text{Al}^{3+}$  substitution falls from 37.42 to 31.35 T, determines the hyperfine field proportional to spontaneous magnetization. The lower magneton number ( $0 \mu_B$ ) of  $\text{Al}^{3+}$  ions, which take the place of the greater magneton number ( $5 \mu_B$ )  $\text{Fe}^{3+}$  ions, is responsible for the drop in the hyperfine field. As (A)-(A), (A)-[B],

and [B]-[B] exchange interactions exist for spinel ferrites, it is important to note here that the (A)-[B] exchange interaction is preferable among the three exchange interactions. In the present study, the (A)-[B] exchange interaction was greatly subsidized by the inclusion of  $\text{Al}^{3+}$  ions into the  $\text{Ni}_{0.65}\text{Zn}_{0.35}\text{Fe}_2\text{O}_4$  matrix. Furthermore, a reduction in particle size can be used to explain this. The collective magnetic excitations are weaker because the particle size is smaller. Our Mössbauer experiments are further supported by the fact that the saturation magnetization in our  $\text{Ni}_{0.65}\text{Zn}_{0.35}\text{Al}_x\text{Fe}_{2-x}\text{O}_4$  reduced with the addition of  $\text{Al}^{3+}$ . The literature [55, 56] presents similar findings. Overall, the superexchange interaction seen in the saturation magnetization and the hyperfine interactions was decreased by  $\text{Al}^{3+}$  replacement.

### 3.13 DC Electrical Properties

Using a two-probe method, the DC resistivity of  $\text{Ni}_{0.65}\text{Zn}_{0.35}\text{Al}_x\text{Fe}_{2-x}\text{O}_4$ , where  $x = 0.0, 0.5$ , and  $1.0$ , was determined. The development of high-frequency devices at the nanoscale depends critically on the DC resistivity. Heat treatments, the kind and concentration of dopants, defects, porous nature of the material, density, and grain boundaries can all have an impact on how the DC resistivity varies. All of the samples DC resistivity plots as  $\log \rho/s 1000/T \cdot^{-1}$  were developed, as shown in Fig. 9. Verwey's hopping mechanism can be used to explain the electric transport mechanism in ferrites. According to this, electron hopping between ions of the same element with different valence states causes conduction in ferrite. The resistivity decreases with rising temperature, as seen by all DC resistivity charts (Fig. 9). An improvement in the mobile charge carriers and an increase in temperature are the primary causes. As a result, the DC resistivity drops as temperature rises. All DC resistivity charts adhere to the Arrhenius law, which is stated as follows [37]

$$\rho = \rho_0 e^{\frac{\Delta E}{kT}} \quad (20)$$

Additionally, Fig. 9 demonstrates that the  $\text{Al}^{3+}$  replacement enhanced the DC resistivity. Since they have a significant affinity for octahedral sites, the  $\text{Al}^{3+}$  sites prevail over them. But it took up space in both octahedral and tetrahedral structures.  $\text{Al}^{3+}$  ions take the place of  $\text{Fe}^{3+}$  ions,



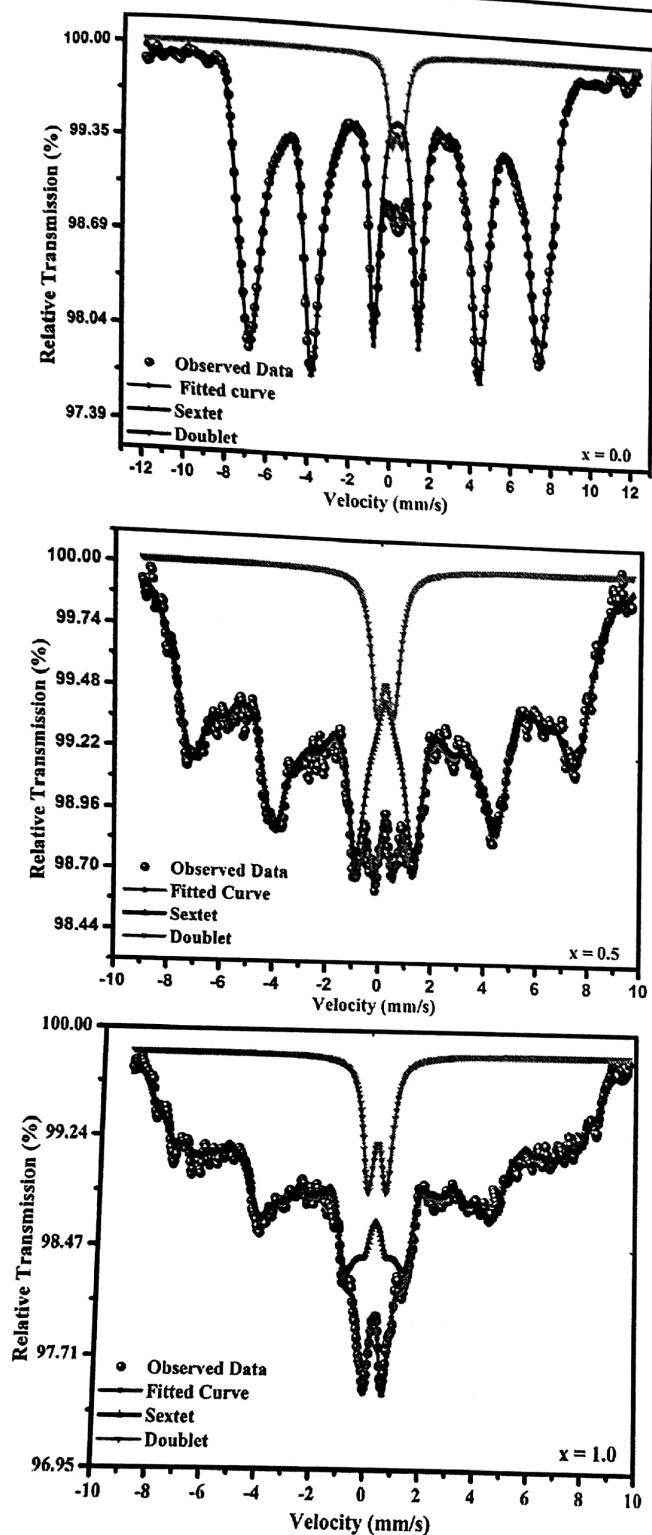


Fig. 8 Mössbauer spectra of  $\text{Ni}_{0.65}\text{Zn}_{0.35}\text{Al}_x\text{Fe}_{2-x}\text{O}_4$ ,  $x=0.0, 0.5,$  and  $1.0$  samples

lowering the amount of  $\text{Fe}^{3+}$  at the octahedral sites. As a result, there are fewer electrons hopping between  $\text{Fe}^{3+}$  and  $\text{Fe}^{2+}$ , which increases resistance. Similar behavior has been seen in [57, 58].

Table 10 The Mossbauer parameters line width ( $\Gamma$ ), isomer shift ( $\delta$ ), quadrupole splitting ( $\Delta$ ) and hyperfine field ( $H_f$ ) of  $\text{Ni}_{0.65}\text{Zn}_{0.35}\text{Al}_x\text{Fe}_{2-x}\text{O}_4$  ( $x=0.0, 0.5$  and  $1.0$ )

| Sample  | Sub Spectrum | $\Gamma$<br>(mm/s) | $\delta$<br>(mm/s) | $\Delta$<br>(mm/s) | $H_f$<br>(T) |
|---------|--------------|--------------------|--------------------|--------------------|--------------|
| $x=0.0$ | S1           | 0.40               | 0.27               | 0.01               | 37.42        |
|         | D1           | 0.50               | 0.29               | 0.43               | —            |
| $x=0.5$ | S1           | 0.43               | 0.19               | -0.01              | 34.45        |
|         | D1           | 0.42               | 0.18               | 0.63               | —            |
| $x=1.0$ | S1           | 0.45               | 0.28               | 0.00               | 31.35        |
|         | D1           | 0.50               | 0.29               | 0.67               | —            |

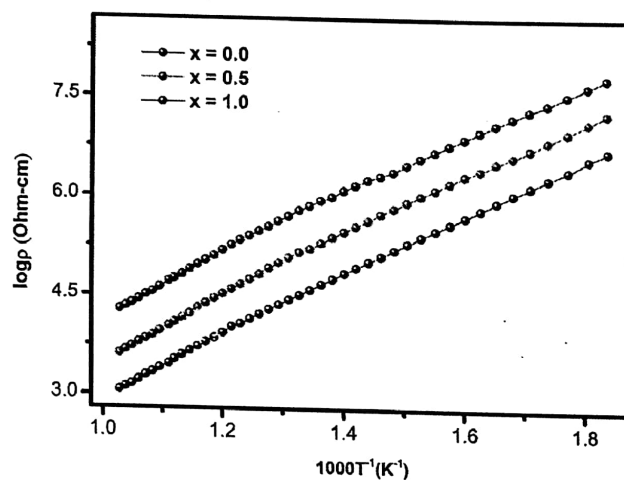


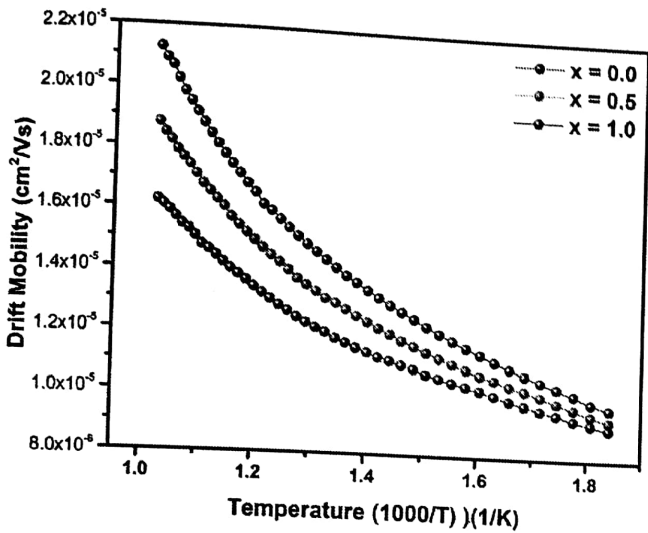
Fig. 9 DC resistivity plots of  $\text{Ni}_{0.65}\text{Zn}_{0.35}\text{Al}_x\text{Fe}_{2-x}\text{O}_4$ ,  $x=0.0, 0.5,$  and  $1.0$  samples

### 3.14 Curie Temperature and Activation Energy

The DC resistivity graphs also show that the curve represents the paramagnetic and ferrimagnetic areas, which are two distinct zones. These areas suggest unique conduction processes with variable activation energies. The paramagnetic region, which follows impurity conduction pathways, corresponds to the high-temperature area. The ferrimagnetic zone follows hopping mechanism and is present in the low temperature area. The Curie ( $T_C$ ) temperature has a significant influence on the phase transition from the ferri-to-para magnetic region. The gradient of the straight line corresponding to the exchange interaction changes as the DC resistivity plot moves through the Curie point. The Curie temperature of that particular composition is determined by this change. Table 11 lists the expected Curie temperature for each sample. It is obvious that  $\text{Al}^{3+}$  substitution lowers the Curie temperature. The weakening of the A-B exchange interaction is responsible for this. The outcomes are comparable to those for

**Table 11** Electrical parameters of  $\text{Ni}_{0.65}\text{Zn}_{0.35}\text{Al}_x\text{Fe}_{2-x}\text{O}_4$  ( $x=0.0, 0.5$  and  $1.0$ ) samples

| Sample  | Activation Energies |            |                 | $n \times 10^{22}$<br>(atoms/cm <sup>3</sup> ) | Tc<br>(K) | D<br>(cm <sup>2</sup> /s) |                      |                      |
|---------|---------------------|------------|-----------------|--|-----------|---------------------------|----------------------|----------------------|
|         | $E_p$ (eV)          | $E_r$ (eV) | $\Delta E$ (eV) |  |           | At                        |                      |                      |
|         |                     |            |                 |  |           | 473 k                     | 723 k                | 973 k                |
| $x=0.0$ | 1.31                | 0.99       | 0.32            | 9.59   | 823       | $9.9 \times 10^{-8}$      | $2.2 \times 10^{-7}$ | $4.4 \times 10^{-7}$ |
| $x=0.5$ | 1.06                | 0.87       | 0.19            | 9.22   | 723       | $9.8 \times 10^{-8}$      | $1.9 \times 10^{-7}$ | $3.7 \times 10^{-7}$ |
| $x=1.0$ | 0.81                | 0.78       | 0.13            | 8.85   | 613       | $9.7 \times 10^{-8}$      | $1.7 \times 10^{-7}$ | $2.9 \times 10^{-7}$ |



**Fig. 10** Drift mobility plots of  $\text{Ni}_{0.65}\text{Zn}_{0.35}\text{Al}_x\text{Fe}_{2-x}\text{O}_4$ ,  $x=0.0, 0.5$ , and  $1.0$  samples

micro spinel ferrite systems described in [59, 60]. For all of the samples, the activation energy was calculated using Eq. 20 and is shown in Table 11. The paramagnetic zone has larger activation energy than the ferrimagnetic region. This is attributed to the ferrimagnetic region's disordered magnetic spin relative to the ordered spin. The difference between the activation energies of the paramagnetic and ferrimagnetic areas is known as the net activation energy. With  $\text{Al}^{3+}$  substitution, a reduction in activation energy is seen. On the basis of a reduction in the lattice parameter with  $\text{Al}^{3+}$  substitution, this can be explained. The interionic distances decrease as a result of the decrease in lattice parameter. As a result, the number of  $\text{Al}^{3+}$  ions increases while the activation energy drops.

### 3.15 Drift Mobility and Diffusion Coefficient

Additionally, the diffusion coefficient and drift mobility were assessed using the following relations [61]:

$$\mu_d = \frac{1}{ne\rho} \quad (21)$$

$$n = \frac{N_A d_b P_{Fe}}{M} \quad (22)$$

$$D = \frac{\sigma k_B T}{Ne^2} \quad (23)$$

where,  $\mu_d$  is the drift mobility,  $n$  is the charge carrier concentration,  $e$  is the charge on the electron,  $\rho$  is the resistivity,  $N_A$  is Avogadro's number,  $d_b$  is the bulk density,  $P_{Fe}$  is the number of iron atoms,  $M$  is the compositions molecular weight,  $D$  is the diffusion coefficient,  $\sigma$  is the conductivity,  $k_B$  is the Boltzmann constant,  $T$  is the temperature, and  $N$  is the number of atoms/cm<sup>3</sup>.

Figure 10 displays the drift mobility graphs as a function of temperature. As the DC resistivity decreases and displays semiconducting characteristics, there can be a noticeable increase in drift mobility with temperature. In addition, it decreased with  $\text{Al}^{3+}$  substitution as DC resistivity increased. This can be accounted by a decrease in charge carrier concentration with  $\text{Al}^{3+}$  ions substitution. Table 11 shows the predicted charge carrier concentration using Eq. 22, which ranges from  $9.59$  to  $8.85 \times 10^{22}$  atoms/cm<sup>3</sup>. Table 11 displays the values of the diffusion coefficient at three different temperatures. The findings showed that the diffusion coefficient increased with temperature while falling with  $\text{Al}^{3+}$  replacement. This is explained by the substitution of  $\text{Al}^{3+}$  ions in the sub-lattice, which results in the production of cation vacancies and a decrease in oxygen vacancies. Drift mobility and diffusion coefficient exhibited behavior that was quite similar, according to findings [58].

### 3.16 Dielectric Properties

By measuring the dielectric constant ( $\epsilon'$ ) and dielectric loss tangent ( $\tan\delta$ ), the dielectric behavior of all the samples was investigated as a function of frequency at room temperature. Dipolar, ionic, electrical, and interfacial polarization play a major role in the dielectric response at the nanoscale. In the low-frequency range, the dipolar and interfacial polarizations predominate.



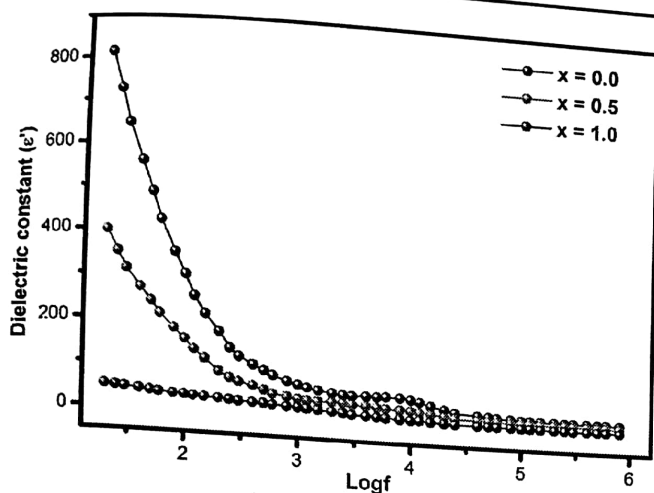


Fig. 11 Dielectric constant as a function of frequency of  $\text{Ni}_{0.65}\text{Zn}_{0.35}\text{Al}_x\text{Fe}_{2-x}\text{O}_4$ ,  $x = 0.0, 0.5$ , and  $1.0$  samples

### 3.16.1 Dielectric constant

Using Eq. 24, the dielectric constant ( $\epsilon'$ ) values were calculated and plotted versus  $\log F$ , as seen in Fig. 11.

$$\epsilon' = \frac{Ct}{\epsilon_0 A} \quad (24)$$

where  $C$  is the capacitance,  $t$  is the thickness of the sample,  $A$  is the area and  $\epsilon'_0$  is the free space permittivity.

Investigations into the variations in dielectric constant were conducted between 50 Hz and 5 MHz. A large dispersion was seen at lower frequencies, and it steadily decreased to a consistent value in the higher frequency range. This illustrates a typical property of the dielectric constant. The electron hopping mechanism, which causes electric dipoles, can explain the observed dielectric behavior. According to the Maxwell–Wagner model of interfacial polarization correlated to cation polarization is connected with the dramatic reduction in the lower frequency range. It is connected to Koop's theory and is exposed to the varied structure made up of the poorly conductive grain boundary. The polarization reduces as frequency increases, which causes the dielectric constant to drop. The applied frequency and the polarization could not be synchronized. As a result, the applied frequency is not kept up with by the electrical exchange between  $\text{Fe}^{3+}$  and  $\text{Fe}^{2+}$ . Moreover, with  $\text{Al}^{3+}$  replacement, the reduced dielectric constant is also noticeable. This is due to the  $\text{Al}^{3+}$  ions being substituted in Ni–Zn spinel ferrite which suppress the production of  $\text{Fe}^{2+}$  and polarization. As a result, the dielectric constant drops that can be explained by the fewer electric dipoles because there is less electron hopping between  $\text{Fe}^{3+}$  and  $\text{Fe}^{2+}$ . According to [62],  $\text{Al}^{3+}$ -substituted Ni–Zn ferrites behaved similarly. It is important to note that the compositional dependence of dielectric constant

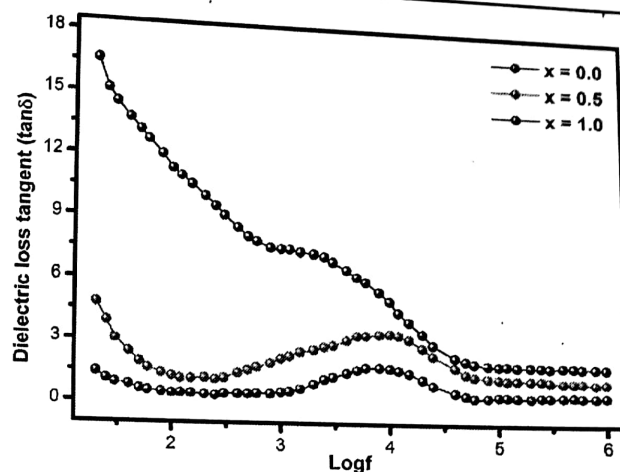


Fig. 12 Dielectric loss tangent as a function of  $\text{Ni}_{0.65}\text{Zn}_{0.35}\text{Al}_x\text{Fe}_{2-x}\text{O}_4$ ,  $x = 0.0, 0.5$ , and  $1.0$  samples

conflicts with the DC resistivity. In the current study, the dielectric constant dropped with regard to the  $\text{Al}^{3+}$  content while the DC resistivity increased. Therefore, the DC resistivity behavior provides strong support for the observed dielectric behavior.

### 3.17 Dielectric Loss Tangent

The energy dissipation in dielectric materials is measured by the dielectric loss tangent ( $\tan\delta$ ), which is dependent on structural homogeneity, stoichiometry, and ferric ion concentration. Figure 12 shows how the dielectric loss tangent varies in relation to the applied frequency. At low frequencies, it drops exponentially and shows a rapid decline, whereas at higher frequencies, it decreases gradually and approaches a nearly constant value. The polarization of cations is thought to be the cause of the abrupt drop in response at low frequency. This can be explained according to the Maxwell–Wagner interfacial polarization model [63], which is based on a heterogeneous structure made up of grains with low conductivity grain borders. Furthermore, it is in line with Koops' theory [64]. It is well known that grain boundaries with good conductivity are active at high frequencies, while those with weak conductivity are more active at low frequencies. Moreover, the dielectric loss tangent at low frequency is maximum if the applied signal frequency is less than the hopping frequency of the ions between  $\text{Fe}^{3+} \leftrightarrow \text{Fe}^{2+}$ . As a result, the electrons obey the field by hopping between  $\text{Fe}^{3+} \leftrightarrow \text{Fe}^{2+}$ , and the maximum loss is seen. As the hopping frequency of ion exchange cannot follow the applied signal at higher frequencies, the minimal dielectric loss tangent is seen. This is the primary cause of both the large dielectric loss tangent seen at low frequencies and the low dielectric loss tangent seen at high frequencies. Thus,



the dielectric loss tangent falls rapidly at low frequency because the electrons hopping frequency cannot match the applied frequency. However, abnormal behavior was seen in the form of peaks at specific frequencies. The observed abnormal behavior of the dielectric loss tangent can be explained by the resonance effect. It occurs when the applied frequency and the frequency at which electrons hopping occur from the  $\text{Fe}^{3+} \leftrightarrow \text{Fe}^{2+}$  transition are almost similar. Peaks are observed that result in power loss when the oscillating ions receive the majority of their energy when the hopping frequency of the charge carriers coincides with the applied frequency of the electric field. The resonance effect in the present study was observed between 5 and 25 kHz. Although it spans the specified frequency range, its highest frequency is around 20 kHz. This occurs when the maximum power is delivered to the oscillating ions. According to [62], trivalent ion-substituted Ni–Zn ferrite systems exhibit a similar loss tangent behavior.

#### 4 Conclusion

Through a solution-gelation process, nanocrystalline  $\text{Ni}_{0.65}\text{Zn}_{0.35}\text{Al}_x\text{Fe}_{2-x}\text{O}_4$  ( $x=0.0, 0.5, \text{ and } 1.0$ ) was successfully prepared. A single-phase cubic spinel structure with the Fd-3 m space group was discovered via Rietveld analysis. The presence of two distinct vibrational frequency bands correspond to tetrahedral and octahedral sites, validated the creation of the spinel ferrite structure using FTIR. Additionally, it was discovered that  $\text{Al}^{3+}$  substitution had a considerable impact on the Debye temperature, bulk modulus, and stiffness modulus assessed using FTIR data. An aggregation of grains and a floppy morphology were visible in the SEM pictures. Additionally, TEM scans showed that the particles were spherical in shape. Neel's sublattice model was used to explain magnetic findings and support their trend with  $\text{Al}^{3+}$  replacement. XRD measurements support the cation distribution predicted from magnetization. Moreover, it was discovered that after  $\text{Al}^{3+}$  substitution, the hyperfine field calculated from Mossbauer had a significant impact. The DC electrical resistivity behavior suggested that the sample were semiconducting. With  $\text{Al}^{3+}$  replacement, the dielectric studies revealed a declining trend. To summarize, these ferrite systems are excellent for high frequency devices due to their high DC electrical resistivity, low dielectric loss, and moderate magnetic characteristics.

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analysis and calculations, writing—original draft, JSK: Cation distribution, Elastic property determination, AK: Mössbauer data curation and analysis, RVK: Conceptualization, Supervision, KMJ—Conceptualization, Validation.

#### Declarations

**Conflict of interest** The authors state that they are aware of no personal or professional conflicts that might have appeared to have impacted the findings provided in this study.

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