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Synthesis, Characterization, Antibacterial and Antifungal Activities of Manganese (II) Complex of (E)-1-(2,6-dihydroxyphenyl)-3-(5-methylfuran-2-yl)prop-2-en-1-one Ligand

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ABSTRACT

TheManganese (II)metal complex has been synthesizedby using novel(E)-1-(2,6-dihydroxyphenyl)-3-(5-methylfuran-2-yl)prop-2-en-1-one ligand. The ligand was prepared by the Claisen-Schmidt condensation method of 2,6-dihydroxy acetophenone and 5-methylfurfural. The structure of the complex has been characterized by the analytical data such asElemental analysis, magnetic moment, conductivity measurement, UV-Vis spectrum, IR spectrum. Analytical data shows 1:2 stoichiometry and the magnetic moment, suggests thatMn(II) complexhasoctahedral geometry.The conductivity data revels that the complex is non electrolyte.Antibacterial and Antifungal activities of the complex with selected bacterial strain and fungal strain carried out and the results have been compared with commercial standards.The synthesized Mn(II) complex shows promising antibacterial and antifungal activity.

Keywords: Antibacterial and Antifungal activities, UV-Vis spectrum, IR spectrum,Physico-chemical property, Magnetic Susceptibility and Conductivity.

I. INTRODUCTION

Chalcones are constitute an important group of natural products, which has two aromatic rings joined by α, β unsaturated carbonyl system. Chalcones are open chain precursors for biosynthesis of flavonoids and isoflavonoids and occur mainly as polyphenolic compounds whose color changes from yellow to orange [1]. They exist as either Trans (E) or Cis (Z) isomers having two aromatic rings those are joined by a three carbon alpha beta unsaturated system. In case the E isomer is more stable from the perspective of thermodynamics, which makes it the predominant configuration among the chalcone. The configuration of the Z-isomer is unstable due to the strong steric effect between the carbonyl group and the A ring [2].

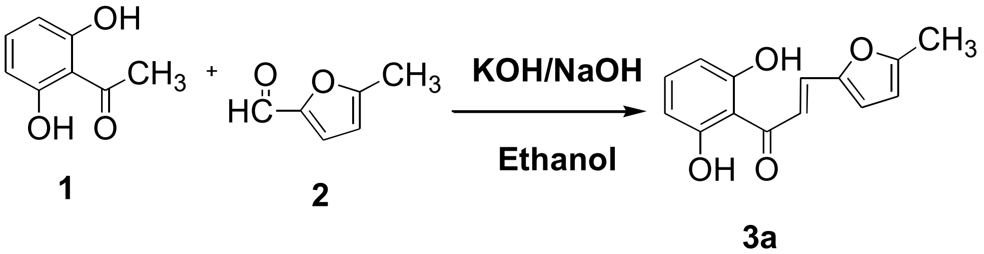
The electrophilic nature of the alpha-beta unsaturated carbonyl system, this moiety is capable of forming the irreversible bond with biological macromolecules, resulting in a number of toxic effects, such as allergenic reactions, carcinogenicity, and mutagenicity[3].

The metal complexes possess interesting biochemical properties, such as antioxidant, antimalerial,antitumor, anti-fungal and antimicrobial activities[4].

II. MATERIALS AND METHODS

2.1 Synthesis of (E)-1-(2,6-dihydroxyphenyl)-3-(5-methylfuran-2-yl)prop-2-en-1-one ligand (3a):

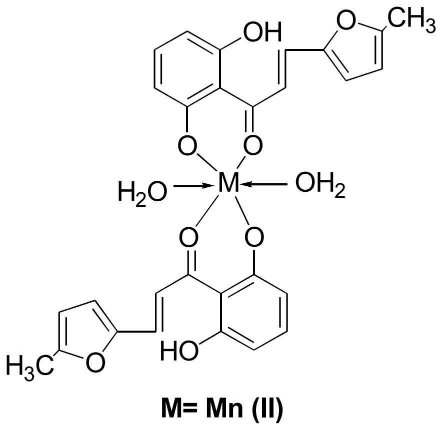
The reagents used for preparation of(E)-1-(2,6-dihydroxyphenyl)-3-(5-methylfuran-2-yl)prop-2-en-1-one (**3a**)are of A.R. grade. The mixture of 2,6-dihydroxy acetophenone (**1**) (0.01 mol) and 5-methyl-Furaldehyde (**2**) (0.01 mol) is dissolved in ethanol (20 mL) and then sodium hydroxide 10 mL (40%) was added to it. The mixture was stirred for overnight till brown color precipitation was observed. The progress of the reaction was monitored by Thin Layer Chromatography by using eluent Petroleum ether: Ethyl acetate (7:3), from thin layer chromatography the completion of the reaction is observed. After completion of the reaction, the contents were poured into ice cold water and then acidified by dil.HCl. The solid obtained was filtered and crude product (**3a**) was recrystallized from ethanol to give the chalcone [5]. (**Scheme-1**)



Scheme-1: Synthesis of (*E*)-1-(2,6-dihydroxyphenyl)-3-(5-methylfuran-2-yl)prop-2-en-1-one Ligand

2.2 Synthesis of Metal Complex:

The solution of 0.02 mole of (E)-1-(2,6-dihydroxyphenyl)-3-(5-methylfuran-2-yl)prop-2-en-1-one (**3a**) ligandwas taken in round bottom flask containing 30 ml of anhydrous methanolicsolution and boiled for 10 minutes. A hot solution of 0.01 mole, ofManganese acetate in 20 ml of methanol was added drop wise to the solution of the chalcone of 5-methylfurfural (**3a**)to this reaction mixture, 10% alcoholic ammonia was added up to slightly alkaline pH. The complex was precipitated atpH 8 range. The pH8-10range was definite for these complexes [6]. The content was stirred on magnetic stirrer for one hour. The solid metal complex separated out and washed with methanol three to four times. The melting point of the synthesized metal complex (**4a**) was determined by Thiele's melting apparatus. The structure of Mn (II) complex is shown in **Figure-1**.



(4a)

Figure-1: Structure of Mn (II) complex (4a)

III. RESULTS AND DISCUSSION

3.1 Physical parameters:

Metal complex of Manganese (II)with (E)-1-(2,6-dihydroxyphenyl)-3-(5-methylfuran-2-yl)prop-2-en-1-one (**3a**)was reddish brown in color. The complex was precipitated atpH 8 range, having Melting point $310^{0}C$. The complex is insoluble in water and soluble in DMSO, DMF [7].

3.2 Magnetic susceptibility, solutionconductivity and electronic absorption spectral data

3.2.1: Magnetic susceptibility:

The magnetic moment of Mn(II) complexes in the present investigation are in the range which is almost close to the spin only value of 5.92 B.M. These values are in good agreement with the moment reported for mononuclear high spin octahedral Mn(II) complexes by earlier workers [8].

Metal complex	**Molar Conductance Ohm^{-1} cm^{2}mol^{-1}**	**μ_{eff} (B.M.)**	**Absorption Maxima cm^{-1} (nm)**		
			$^{6}A_{1g} \rightarrow {}^{4}T_{2g}$ (G)	$^{6}A_{1g} \rightarrow {}^{4}A_{1g}$ (G), $^{4}E_{g}$	Charge Transfer
Mn(II) Complex	4.37	5.86	24630(406)	29411(340)	33557(298)

3.2.2: Solutionconductivity and electronic absorption spectral data:

The solution conductivities of 10^{-3} M solution of metal complex in DMSO were measured on EQUIPTRONICSdigital conductivity meter EQ - 660 with 20 μΩ to 200 μΩ at 298K temperature. They are insoluble in water and soluble in DMSO, DMF. The low solution conductivity of 10^{-3} M solutions of Mn(II) complexes in DMSO indicates their non-electrolytic nature.

Figure-2: Electronic absorption spectrum of synthesizedMn (II) complex

The electronic absorption spectra of Mn(II) complexes were showed three bands at 19,120 to 25000 cm^{-1}, 25125 to 27700 cm^{-1}, and 28993 to 30581 cm^{-1} assignable to $^6A_{1g}\rightarrow{}^4T_{2g}$(G), $^6A_{1g}\rightarrow{}^4E_{1g}$ or $^6A_{1g}\rightarrow{}^4T_{1g}$ (G) and charge transfer indicating octahedral geometry around the metal ion [9-10]. (**Figure-2**).

3.3 CHO analysis of synthesized complex (4a):

The carbon, hydrogen, oxygen, Manganese metal percentage in Mn (II) complex of chalcone measured at SAIF Cochin,Kerala. The calculated and measured values of CHO analysis are matching and are given in the **Table-1**.

Table-1: CHO analysis of synthesizedMn (II) complex

Metal complex	**Chemical formula**	**Mol. Wt.**	**Elemental analysis :% found (calculated)**			
			C	H	O	M
Manganese (II)	[$C_{28}H_{26}O_{10}Mn$]	577	58.23 (65.50)	4.53 (5.10)	27.70 (18.70)	9.51 (10.70)

3.4Infra-red spectrum:

The IR spectrum of α, β-unsaturated carbonyl group has characteristic bands of chalcone at prominent bands between 1625 to 1650 per cm [11-12]. The characteristic peaks in infra-red spectrum give the presenceof particular functional group. The region at which other absorption bands appear depends on the type of aromatic / hetero-aromatic rings as well as the substituent present on these rings. The infrared spectrum of metal complex of Manganese (II)with (E)-1-(2,6-dihydroxyphenyl)-3-(5-methylfuran-2-yl)prop-2-en-1-one was recorded on a Perkin- Elmer Spectrum RX-IFTIR Spectrophotometer in the range 4000-400 cm^{-1}(**Table-2**) using potassium bromide pellet at CIL, Chandigarh, Punjab. The stretching frequency of metal complex of Manganese (II)with(E)-1-(2,6-dihydroxyphenyl)-3-(5-methylfuran-2-yl)prop-2-en-1-one is represented in table number (2) and the IR spectrum in **Figure-3.**

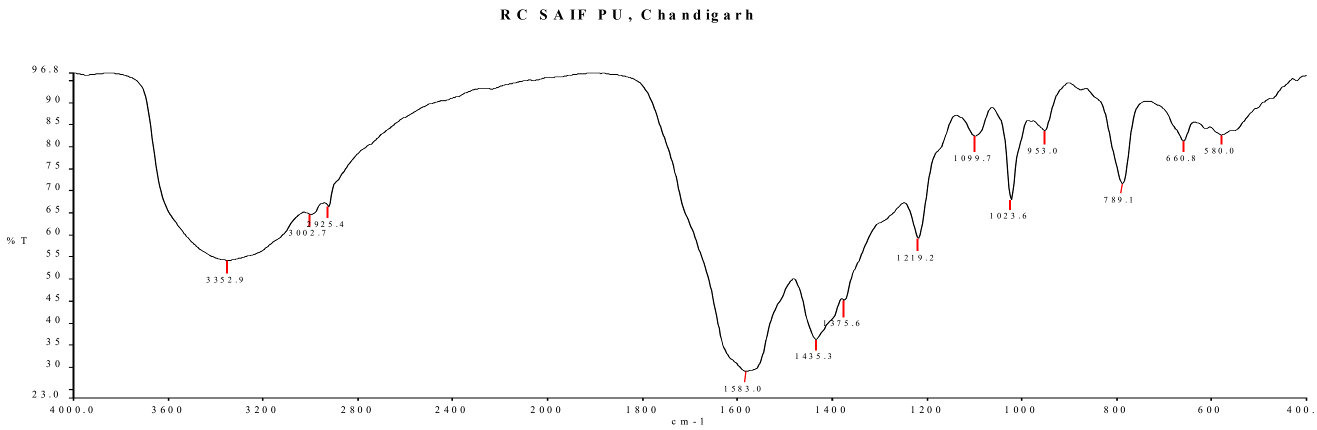


Figure-3: IR spectrum of metal complex of Mn (II)with(E)-1-(2,6-dihydroxyphenyl)-3-(5-methylfuran-2-yl)prop-2-en-1-one Ligand

Table-2: IR spectral data ofMn (II) complex with(E)-1-(2,6-dihydroxyphenyl)-3-(5-methylfuran-2-yl)prop-2-en-1-one Ligand:

Ligand/ Metal complexes	**υ (OH) cm^{-1}**	**υ (H$_2$O) cm^{-1}**	**υ (-CO-CH=CH-) cm^{-1}**	**υ (-C=O in pyron ring) cm^{-1}**	**υ (C-O-C) cm^{-1}**	**υ (C=C) cm^{-1}**	**Aromatic Ring (C=C) cm^{-1}**	**υ (M-O) cm^{-1}**
Chalcone	3420	-	1623	-	1095	1558	1439	-
[Mn(A$_2$)$_2$]	3003	3353	1583	-	1023	1435	1375	580

In chalcone ligand, there is a presence of phenolic -OH is confirmed by peak at 3420 cm^{-1} in the spectra of Mn(II) complexes, that is the complete disappearance of the peak at 3420 cm^{-1} suggests absence of phenolic group -OH indicates its coordination. (C-O-C) is shifted to a lower wave number compared with the free ligand. The new band is observed at 3352 cm^{-1} which is due to the -OH intense broad band of coordinated water molecules. In Mn(II) complexes new band is observed at 580 cm^{-1} due to the (M-O) bond. The band assigned to the carbonyl group (1623 cm^{-1}) is shifted to a lower wave number compared with that of free ligand, (1583cm^{-1}) proving its coordination. In the spectra of Mn(II) complexes, the strong bands appeared in the range of 1435-1490 cm^{-1} can be assigned to (C=C) stretching vibrational mode. These bands are found to remain unchanged in the complexes from their corresponding ligands, which indicate the non-involvement of (C=C) group in complex formation. The bands due to (C=C) aromatic ring vibrations may remain the same or shift their position as a result of change in the distribution of electrons and molecular environment due to introduction of a metal ion.

Omar-Al-Obaidi was assigned in flavone that, In the FT-IR spectra of ligands, the presence of phenolic –OH and the carbonyl group is confirmed by peaks at 3401 cm^{-1} and 1600 cm^{-1} however in the spectrum of the complexes that is the complete disappearance of the peak at 3401 cm^{-1} suggests absence of phenolic group –

OH which indicates its coordination. The band assigned to the carbonyl group is shifted to a lower wave number comparing with that of free ligand, proving its coordination[13].

3.7Antibacterial and Antifungal Activity:

3.7.1: Antibacterial Activity: The antibacterial activity of Mn (II) complex was studied, for evaluating antibacterial activity, Gram positive and Gram negative bacterial pathogens were used. *Staphylococcus aureus*ATCC 6538*,* *Bacillus megaterium*ATCC 2326, *Bacillus subtilis* ATCC 6633 were Gram positive pathogens used in this study. *Escherichia coli* ATCC8739*, Salmonella typhi*ATCC9207*, Shigellaboydii*ATCC 12034*, Enterobacteraerogenes*ATCC13048*, Pseudomonas aerogenosa*ATCC9027, *Salmonella abony* NCTC6017 were the Gram-negative pathogens used in this study.

3.7.2: Antifungal Activity: Antifungal activity was assayed by cup plate agar diffusion methodby measuring inhibition zones in mm. *In vitro*antifungal activity of synthesized compound and standard have been evaluated against strains of the fungal toxicity ofMn (II) complexwas studied *in vitro* against *Aspergillusniger*ATCC 16404*, Saccharomyces cerevisiae* ATCC 9763, *Candida albicans* ATCC10231 fungal pathogens at fixed 1% concentration.

From the result of antibacterial and antifungal activities of ligand and complex, it is clear that the complex shows enhanced activity than ligand. The increase in antimicrobial activity is due to faster diffusion of metal complexes as a whole through the cell membrane or due to the combined activity of the metal and ligands [14].

IV. CONCLUSION

TheMn (II) complex was colored, soluble in most of the organic solvent. The stoichiometryratios of the metal complexwasobtained has been found to be 1:2.Solution conductivity of this metal complexshows non-electrolytic nature. The CHO analysis gives C, H, and O percentage in the metal complex. The magnetic moment, UV and IR spectral data suggests that Mn (II) has Octahedral geometry.From the antibacterial and antifungal activities of ligand and complex, it is clear that the complex shows enhanced antibacterial and antifungal activities than ligand.

V. ACKNOWLEDGEMENT

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Review on Biomedicinal Applications of Ferrite Nanoparticles

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ABSTRACT

Ferrite Nanoparticles exhibit enormous promise in biological applications due to their capacity to accurately regulate the behaviour by an external magnetic field. Ferrite Nanoparticles are remarkable magnetic capabilities make them ideal nanoagents for a variety of applications such as targeted medication administration, MRI, biosensors, magnetic hyperthermia (MHT), magnetic separation and antibacterial agents. The issue is maintaining the high magnetism, which declines when size is reduced to the nanoscale.The manufacture of these ferrite nanoparticles is significant, with the selection of an optimum synthesis procedure playing an important role.As a result, characteristics like as shape, chemical and physical properties, and biocompatibility influence the effectiveness of ferrite nanoparticles in biomedical field. In this review article, we focuse the biological importance of Ferrite Nanoparticles.

Keywords: MRI; superparamagnetism; targeted drug delivery; magnetic hyperthermia; nanoferrites; toxicity.

I. INTRODUCTION

In past few years, nano-science, nanotechnology and nanostructurehave now become common words not only in science but also in daily life. Nano-materials depict, in principle, materials of which a single unit is sized (at least one dimension) between 1-100 nm (10 meter) can be accomplished by the assembly of the constituents of molecules or atoms of few angstroms or 10^{-10} m in size. Nano-materials study takes a materials science based approach to nanotechnology in materials measurement and synthesis which have been developed in support of micro-fabrication research. Materials with arrangement at the 'nanoscale' (1-100 nm) often have unique electronic, optical, mechanical properties, which are expected to be utilized in several different applications, including optical filters, sensors, low-threshold laser, biological detection and controlled drug delivery.

The research on synthesis, exploration and characterization of nanosized (1-100nm) materials were increased in the last few decades for the development of science. The term "nanoscale materials" refers to a group of substances with at least one dimension of fewer than 100 nanometers[1]. Some nanoparticles are found naturally, but artificial nanomaterials are of particular interest. Engineered nanomaterials are resources that have been developed at the molecular (nanometre) level to make use of their small size and unique features that aren't found in their bulk counterparts[2]. Increased relative surface area and novel quantum effects are the two main reasons why materials might have distinct properties at the nanoscale. Nanomaterials have a substantially higher surface area to volume ratio than traditional materials, which can increase chemical reactivity and weaken them[3]. Quantum effects can also play a greater role at the nanoscale in determining material properties and characteristics, resulting in unique optical, electrical, and magnetic features.

As a result, nanomaterials have important applications such as biosensors, medication delivery, energy storage, and so on. On the basis of dimensions, nanomaterials are classifieds into four different classes. The properties of nanomaterials depend on the synthesis procedure. Basically, nanomaterials are synthesized by using two different approaches, one is a top-to-down approach and the second is the bottom-up approach.

Ferrites have established their potential in several applications due to their remarkable electrical and magnetic properties and also in magnetic resonance imaging (MRI). Nanomaterials are interesting because they exhibit unique optical, magnetic, electrical, and other properties at such a small scale. These emergent features have the potential to have huge implications in sectors like electronics, medicine, and others. In comparison to bulk materials, nanoparticles exhibit significantly innovative and improved chemical, physical, magnetic, and other properties[4].

II. INTRODUCTION TO FERRITES

Ferrite exhibits ferrimagnetisms due to the super-exchange interaction between electrons of metal and oxygen ions. The opposite spins in ferrite results in the lowering of magnetization compared to ferromagnetic metals where the spins are parallel. Due to the intrinsic atomic level interaction between oxygen and metal ions, ferrite has higher resistivity compared to ferromagnetic metals. This enables the ferrite to find applications at higher frequencies and makes it technologically valuable. Ferrites are chemical compounds consisting of ceramic materials with iron oxide (Fe_2O_3) as their main component [5]. The chemical formula of ferrite is generally expressed as $MeFe_2O_4$, where 'Me' represents a divalent metal ion. (e.g. Fe^{2+}, Ni^{2+}, Mn^{2+} , Mg^{2+}, Co^{2+}, Zn^{2+}, Cu^{2+}etc). The crystal lattice of ferrite is spinel with cubic symmetry.

The crystal structure of spinel ferrite possesses two sub-lattices namely tetrahedral (A) and octahedral [B] sites. The cations can occupy either tetrahedral (A) or octahedral [B] or both sites partially. On the occupancy of cations at tetrahedral (A) and octahedral [B] sites, the spinel ferrites are known as normal, inverse and random spinel. Most important commercial spinel ferrites are cobalt ferrite and nickel ferrite have inverse spinel structureand they exhibit high electrical resistivity, high saturation magnetization, high permeability and high Curie temperature[6]. The high values of resistivity and magnetic properties are useful in making composites materials.

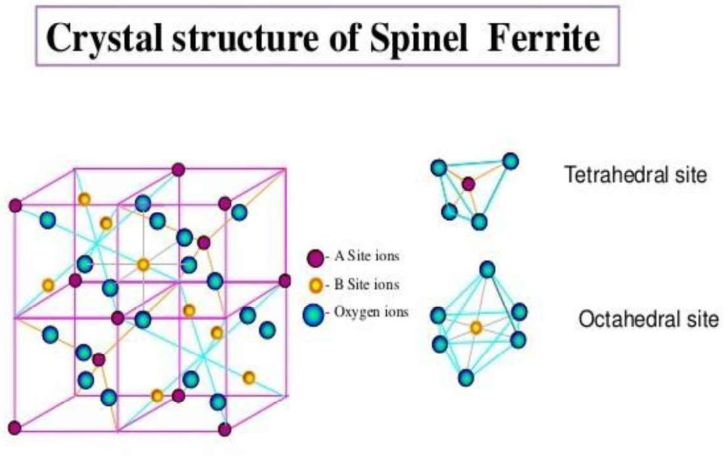

Figure 1: Crystal structure of spinal ferrite.

Among the magnetic ceramics, magnetic oxides are the most Important and rather the only relevant material from the point of view of their application .The number of mixed oxides such as ferrites, ferroelectrics, peroviskites etc, are being used in many technological applications. Ferrite are mixed metal oxide having iron oxide as their main constituent. Ferrite can be classified into three group's viz. spinal ferrite, garnet and magnetoplumbite according to their crystal structure. Spinal ferrite has the general formula MFe_2O_4, Where M is divalent metal ions like Co, Ni, Mg, Mn, Zn, Cd, Fe etc. Ferrites are the ferromagnetic oxides with combined electric and magnetic properties, which are useful in many applications such as antenna rod, transformer core, memory chips, sensors ,as catalyst, microwave devices like circulators, phase shifters, gyrator etc. Apart from their excellent elliptical and magnetic properties they have another advantage like chemical stability, easy preparation, low cost etc. They exhibit high electric resistivity, low eddy current and dielectric losses, high saturation magnetization, high curie temperature etc. These properties of ferrites are sensitive to method of preparation, cation distribution, type and nature of dopants also radiation dose, dose rate etc.

Types of ferrites

Types	Structure	General formula	Example
Spinal	Cubic	$M^{II}Fe_2O_4$	M^{II}=Cd,Co,Mg,Ni,Zn etc.
Garnet	Cubic	$Re^{II}Fe_2O_{12}$	Re^{II}=Y,Sm,Eu,Cd,Tb,Dy,etc.
Magnetoplumbite	Hexagonal	$Me^{II}Fe_2O_{19}$	(Er,Tm and Lu)

Ferrites are ferromagnetic in structure as originally proposed by Neel.It play an important part in the field of telecommunication, electronic, entertainment digital computer industries. Since the appearance of the first commercial ferrite product was in about 1945. The unique property of these ferrites is high magnetic permeability and high electrical resistivity. The required properties of ferrite may be classified as intrinsic and extrinsic. Number of applications depends on the extrinsic properties.

Nickel ferrite

Nickel Ferrite is a significant magnetic material with a wide range of applications, including ferrofluid fabrication, catalysis, and magnetic refrigeration. It is one of the most important soft ferrites, with low conductivity, low eddy current losses, and excellent chemical stability, mechanical hardness and electrochemical consistency[7]. Additionally, nickel ferrite's bulk phase is entirely made up of inverse spinel structures. The structure and morphology (form, size, and surface topology) of ferrite nanostructures may be precisely controlled by modifying the composition as well as the techniques of synthesis, according to the researchers. Various procedures for creating ferrite nanostructures have been developed over the years, including solid state, sol-gel, thermal decomposition, co-precipitation, hydrothermal, and mechanical milling. Despite several concerted attempts, a highly efficient and precise process for synthesis of ferrite nanostructures is still a long way off. Furthermore, no association has been discovered between the compositions, cationic distributions in the structure, electric and magnetic characteristics of ferrite nanostructures. This ferrite is an inverted spinel ferrite, with eight units of $NiFe_2O_4$ incorporated within the spinel structure's cell. Half of the ferric ions preferentially occupy tetrahedral sites, whereas the other half preferentially occupy octahedral sites. As a result, the compound is represented by the formula $(Fe^{3+})_A\ [Ni^{2+}Fe^{3+}]_BO_4^{2-}$.

The magnetic characteristics of nickel ferrite are comparable to those of magnetite and maghemite. Composition and microstructure, which are susceptible to the synthesis preparation technique, have an impact on the characteristics of synthesised materials. Furthermore, the magnetic properties of $NiFe_2O_4$ nanoparticles are very size-dependent. The creation of $NiFe_2O_4$ nanoparticles with the best magnetic characteristics is already generating a lot of attention. Numerous techniques have been devised to create nanocrystalline nickel ferrite, including sol-gel, organic gel-thermal decomposition, hydrothermal, co-precipitation, gel assisted hydrothermal routes, thermolysis, wet chemical co precipitation approach, microemulsion, and microwave synthesis [8]. Nickel ferrite nanoparticles are suitable for photocatalytic and waste water applications due to their large band gap and modern saturation magnetization. The performance of devices utilised in sensing and biological applications, such as contrast agents for magnetic resonance imaging and heat mediators for magnetic fluid hyperthermia, may be implemented due to the large tuning ability of nickel ferrite[9].

The following are the main applications of nickel iron oxide nanoparticles:

- Resistant suspension for levitated railway systems
- In the process of preparing nickel cermet for the anode layer of solid oxide fuel cells
- Magnetic recording medium with a high density
- Magnetic refrigeration
- As a catalyst, magnetic liquid, and microwave absorbers
- In lithium nickel iron oxide cathodes for lithium micro batteries
- In electrochromic coating, polymers, and fabrics.

III. LITERATURE REVIEW

The co-precipitation process was used to make nickel ferrite nanoparticles. To identify the structure of $NiFe_2O_4$ nanoparticles, an X-ray diffraction pattern was employed. FT-IR analysis revealed the existence of $NiFe_2O_4$ nanoparticles. Scanning Electron Microscopy was used to examine the surface morphology of $NiFe_2O_4$ nanoparticles. Transmission Electron Microscopy might be used to measure the particle size of nickel ferrites nanoparticles. The research sought to investigate the dielectric characteristics of nickel ferrite nanoparticles at various frequencies and temperatures, such as dielectric loss and dielectric constant. The magnetic characteristics of nickel ferrites were also investigated. XRD confirmed that $NiFe_2O_4$ nanoparticles corresponded to the cubic spinel structure. $NiFe_2O_4$ nanoparticles have an average particle size of 28nm. The dielectric investigations revealed that frequency had a detrimental influence on both the dielectric constant and the dielectric loss[10].

The $NiFe_2O_4$ nanoparticles were manufactured by the hydrothermal technique and the inhibition of surfactant on the particle growth is explored. It demonstrated that the products were pure $NiFe_2O_4$ and also nanoparticles develop with increasing the temperature, whereas surfactant hinders the particle growth under the same circumstances. The average particle size was evaluated by the TEM micrographs and found to be in the range of 50-60 nm that reduced up to 10-15 nm in the presence surfactant. Nano sized nickel ferrite particles were produced with or without surfactant aided hydrothermal techniques. The findings revealed that with rising in the temperature, the crystallinity of nanoparticles is enhanced. In the presence of surfactants, the crystallinity of $NiFe_2O_4$ nanoparticles reduced in comparison with surfactant - free produced samples. All of the nickel ferrite nanoparticles were superparamagnetic at normal temperature[11].

$NiFe_2O_4$ was synthesized by two methods – chemical sol gel method and the high frequency plasma chemical synthesis and magnetic properties, crystalline size, specific surface area of synthesized products was characterized. The average particle size of nano powders obtained by the sol-gel method self-combustion method is in the range of (25-40) nm and ferrites synthesized in plasma have wider particle size distribution range (10-100) nm with some particles of 200 nm. The magnetic properties of sample obtained by the sol gel self-combustion method differ from those of the plasma products. Dense material from the plasma nano powders forms at 1000 degree Celsius but from the sol gel self-combustion nano powders at 1200degree Celsius[12].

The citrate precursor technique was used to create $Ni_{0.5}M_{0.5}Fe_2O_4$ nanoparticles. At 400 degrees Celsius, the citrate precursor was annealed. An X-ray diffractometer was used to characterise the annealed powders. The observed XRD data was further investigated, revealing that particles annealed at temperatures up to 450 degrees Celsius had a cubic spinel structure, but particles produced at temperatures above 450 degrees Celsius have a tetragonal spinel structure. Sharp variations in particle size, lattice constant, magnetism, and retentivity were detected in the temperature range 450-500 degrees Celsius, indicating that growth differs at temperatures above and below a critical temperature in this range. The characteristics of nano size ferrite samples are highly dependent on the temperature of production[13].

$NiFe_2O_4$ nanoparticles were synthesized using co-precipitation method. The X-Ray diffraction was used to determine the structure of nickel ferrite nanoparticles. The fact that $NiFe_2O_4$ nanoparticles belonged to cubic spinal structure was established by XRD. The presence of nickel ferrite nanoparticles was confirmed by FT-IR spectrum. The details of surface morphology of $NiFe_2O_4$ nanoparticles were obtained by scanning electron microscopic analysis. SEM analysis showed that nanoparticles agglomerated to form spherical-shaped particles. The particle size was determined by Transmission electron microscopy. The average particle size of $NiFe_2O_4$ nanoparticles was found to be 28 nm. The work aimed at investigation of the dielectric properties such as dielectric loss and dielectric constant of $NiFe_2O_4$ nanoparticles at varied frequencies and temperature. From the dielectric studies it became evident that the frequency negatively impacted both the dielectric constant and the dielectric loss as decreased with increase in the frequency[14].

IV. SYNTHESIS METHODS

SOL-GEL TECHNIQUE

The sol-gel method of synthesizing nanomaterials is very popular and is widely employed to prepare ferrite materials[15-16]. The interest in this synthesis technique arises due to the possibility of synthesizing nano ferrite materials at very low temperatures; precursors are very simple chemical reagents, good stoichiometric control, special instruments are not needed, dopant ions can be quickly introduced into the resultant material and less agglomeration of grains in the product. The main advantage is one can get uniform nanoparticles with good quality by the sol-gel approach. The chemical reaction is carried out by dissolving the citric acid and metal nitrates in a minimum quantity of distilled water at room temperature. The citrate precursors are usually preferred due to their low solubility and low decomposition temperature. Generally, in order to achieve a complete reaction within the shortest time period and at the lowest possible temperatures, mixing of the component cations on an atomic scale is necessary. The mixture is heated in order to evaporate excess amount of water. Then the gel was dried at 150^0Cand as a result of which, the dry gel self-ignited to form intended ferrite powder. Throughout the auto-combustion process, exothermic redox chemical reaction along with fuel oxidation and decomposition of nitrates take place. The gasses like N_2 and CO_2 emerge, supporting the formation of nano ferrite powder.

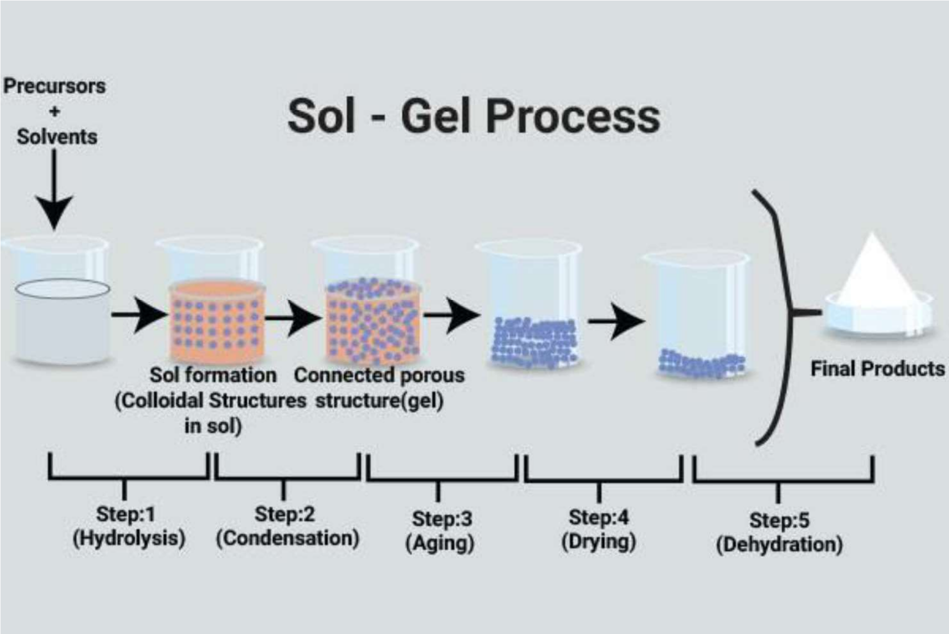

Figure 2. Sol-gel technique

The advantages of this technique are as elucidated below[17-18].

- The intimate mixing of the elements in gel form results in reaching the ingredients in a short period of time.
- Grinding procedure is not required to achieve homogeneous single phase ferrites, which avoids contamination (high product purity).
- The precise size control of the particles is also strength of a sol-gel processing.
- The possibility of controlling the porosity of the product material is highly attractive. (High crystalline ferrite nanoparticles)
- It is attributed to more uniform phase distribution in multicomponent systems like ferrites.

- Exothermic decomposition of precursor provides heat to the solid-state reaction, thus reducing the external temperature required for the ferrite formation in shorter time.
- Fine particle size and uniform particle size distribution
- Simple equipment and simple synthesis process
- It is a cost effective method.

Among the available chemical methods, sol-gel is a simple technique without much- complicated procedure and produce ferrite nanoparticles of high purity at minimum cost.

Solid-State Technique

The solid-state approach is extensively utilised for the production of single crystal and polycrystalline powders of transition metal phosphates and arsenates and monovalent cations[19]. This is a typical method for producing polycrystalline material from solid reagents. This approach use a chemical reaction to generate a new solid with a well-defined structure from solid starting ingredients. Fine grain metal compounds are mixed, pelletized, and heated at a given temperature for a certain amount of time. Extreme circumstances, such as high temperature and pressure, are required for some metal compounds, such as metal oxides or salts, to begin reactions in a molten flux or a rapidly condensing vapour phase. This method is also known as "shake and bake" or "heat and beat" chemistry. Characterizing the reaction rate in solid state synthesis is especially critical. Because approaches for purifying produced solids are extremely constrained, solid-state processes must be completed. The rate of the solid-state reaction is determined by the reaction circumstances, which include the structural qualities, shape, and surface areas of the reactants, the rate of diffusion, and the thermodynamic parameters of the process. The chemical precursors and preparation procedures determine the chemical and physical characteristics of the final products. Metal compound reactions are initiated by an energy source and propagated by the heat emitted during the synthesis of products and byproducts in this approach. The downsides of this approach are its slowness and high energy requirements. In reality, the reaction takes place at high temperatures (500-2000 degrees Celsius) for several hours and continues for several days. Heating at these temperatures may cause the target chemical to breakdown[19].

Hydrothermal technique

The hydrothermal technique is a novel approach for producing nanocrystalline inorganic materials. It is characterised as a homogeneous or heterogeneous reaction that occurs under high temperature and pressure in the presence of aqueous solvents, resulting in the development of solid compounds that are generally insoluble under normal circumstances. It is a low-cost and environmentally friendly approach. Temperature, water, pressure, and reaction time are therefore the three required physical parameters in hydrothermal processing. This synthesis method is also known as a solvothermal procedure, which indicates that additional solvents are employed in addition to water. In a typical experiment, the precursor material is dissolved in a suitable solvent and transported to an autoclave, which is a stainless-steel tank. In such experiments, water is frequently selected as the solvent. After the transfer is complete, the vessel is firmly sealed and exposed to heat treatments. For the synthesis of simple nanostructures, the temperature is set between 80 and 250 degrees Celsius. The supercritical solvent interacts with the precursor material at these extreme temperatures, resulting in the creation of products. Surfactants, in addition to precursor materials, are used in such reactions to create a nanostructure with the appropriate shape. Furthermore, the technique of simplicity and beauty is being forgotten[20]. The hydrothermal approach was used to create aluminium phosphate nanosheets[21].

Application of nanoferrites in biomedical sciences

Ferrite nanoparticles notably ferrites have attracted a lot of interest in recent years due to their uses in various sector and particularly in biomedical field where there improved magnetic characteristics enable diversity in imaging diagnosis and therapy. There are several varieties of ferrites that have been synthesized and presented for diverse uses but ferrite based on cobalt, nickel, and zinc has shown potential for biomedical applications due to strong magnetic biocompatibility and anisotropy[22]. With the objective of increasing the response in magnetic hyperthermia therapies and other biomedical applications, a nanoparticle system based on nickel ferrites has been studied.Monodispersed ferrites nanoparticles with varied proportions of Ni^{2+} ions and sizes have been created using an improved synthesis based on the thermal decomposition method and the seed-growth methodology. It has been demonstrated that structures with low Ni^{2+}cation percentages promote saturation magnetization levels and a decrease in the magnetic anisotropy constant[23]. Recently, magnetic nanoparticles (MNPs) have demonstrated significant promise in biological applications.

Among other uses, MNPs can be utilised as protein separators, contrast agents for magnetic resonance imaging (MRI), medication delivery systems, and heat generators. MNPs have a wide variety of uses and may be utilised for both treatment and diagnostic at the same time. To have a satisfactory performance for both purposes, the qualities and characteristics of the magnetic material should be adjusted (diagnosis and treatment). In the scientific literature, there are several publications on the use of MNPs as heat producers in magnetic fluid hyperthermia or contrast agents for MRI diagnosis or cancer therapy. Despite the fact that both uses are the consequence of magnetic fields interacting with one another[24].

Targeted drug delivery

The low toxicity and good biocompatibility of magnetic nanomaterials form them suitable materials for targeted drug delivery. Magnetic nanoparticles are widely used in targeted drug delivery applications. The direct intake of medicine affects non-targeted healthy organs to overcome these problems targeted drug delivery becomes a standard and promising technique nowadays. The other healthy tissues are not affected by any medicine in the case of targeted drug delivery application[25-26]. Magnetic nanoparticles have excellent magnetic properties which reveal the reduction of nanomagnets concentration in blood. The coating and doping of suitable components increase the effectiveness of magnetic nanoparticles in drug delivery applications.Spinel ferrites nanoparticles have received a lot of interest for their use as a drug delivery agent under the control of an external magnetic field because of their simplicity, effectiveness, ease of manufacture, and capacity to adjust their characteristics for specific biological applications[27–28]. German scientist Paul Ehrlich (1854–1915) introduced the idea of a "magic bullet" that would only kill sick tissue around a century ago[29]. The current history of drug delivery began to use microencapsulated drug particles in the 1950s[30]. The volume of papers in this biological field has significantly expanded since that time.

The literature now accessible contains several studies and research papers employing nanoparticles made of silica[31-32], gold, polymers, and other materials. It follows that since a variety of nanoparticles can be employed, magnetism isn't a need when building a drug delivery nano system. However, the application of magnetic nanoparticles in medication delivery has received significant interest recently[33]. The typical drug delivery approach from non-specific cell and tissue distributions with metabolic instability results in total body toxicity and decreased therapeutic effectiveness[34]. Spinel ferrite nanoparticles' capacity to enclose cytotoxic medicines within the polymer matrix and distribute them to cells is another noteworthy characteristic[35–36]. Spinel ferrite nanoparticles may transport medications without spilling and can quickly reach the target tumour location with the aid of an external magnetic field, helping to successfully treat cancer cells while avoiding normal cells[37]. Up till it reaches the sites of action, the medicine will be released and have therapeutic effects. Multifunctional nanoparticles, which contain semiconductors (for cell imaging), anticancer medicines, spinel ferrite nanoparticles, and biocompatible coating agents, are very useful for cancer therapy. It has been established that $CoFe_2O_4$ nanoparticles with lauric acid caps might be exploited as a potential drug delivery agent with pH-sensitive release[38]. Spinel ferrite nanoparticles may also be made more stable and have less hazardous effects on cells by being coated or capped with biocompatible materials. Spinel ferrite nanoparticles need to be completely demagnetized after being exposed to an external magnetic field in order to be employed for magnetically driven medication delivery.They can also maintain colloidal stability and avoid aggregation as a result, which enables them to be employed in biological applications[39]. One of the hypothesised causes of spinel ferrite nanoparticle aggregation is magnetic attraction between the particles[40].

Magnetic hyperthermia

Magnetic hyperthermia is one of the promising cancer treatments based on the heating ability of nanoparticles. Chemotherapy, radiotherapy, etc are some cancer treatments but these treatments have some drawbacks like these treatments affect normal tissues[41]. To overcome this problem, the use of magnetic hyperthermia is widely increased in the biomedical field. In this treatment, only cancer tissues of cells are killed with the help of an applied magnetic field and do not affect normal tissues or cells. The specific absorbance rate (*i.e.*SAR) value reveals the heating rate of nanoparticles[42]. Hyperthermia, a mild increase in temperature to 40-43 degree Celsius, can trigger the death of cancer cells and boost the effects of radiation and chemotherapy. However, the fulfilment of its full potential as a therapeutically relevant therapy approach was limited due to its inability to heat malignant cells efficiently and locally. This obstacle can be avoided by the intravenous introduction of magnetic nanoparticles focused at cancer cells that aggregate in the tumour, followed by the use of an alternating magnetic field to increase the temperature of nanoparticles situated in the tumour tissue. This tailored approach allows locally heating cancer cells, at the same time, without injuring surrounding normal tissue, which potentially boosts the effectiveness and safety of hyperthermia. The most common materials for magnetic hyperthermia are magnetite or maghemite nanoparticles.

Magnetic nanoparticles can be administered to the tumour by intra-tumoral, intra-arterial, intra-cavitary, intravenous injection. There oral delivery is not possible since most of the nanoparticles will be excreted from the body. Intra-tumoral and intra-cavitary injection localises magnetic particles in the tumour and can lead to efficient heating of primary tumours. Although the following routes of administration are well suited for certain circumstances, intravenous injection is the most adaptable delivery strategy for a wide spectrum of oncological disorders. When magnetic particles of iron oxide are given in this way, the accumulation of nanoparticles of tumour partially depends on the impact of increase permeability and retention[43]. This phenomenon refers to the tendency of nanoparticles to mainly collect in tumours due to the permeability of their vasculature and inadequate lymphatic drainage. Target ligands (antibodies and their fragments, ligands of particular receptors located on the surface of tumour cells, peptides, and aptamers) linked with the magnetic particles might improve the absorption of nanoparticles by malignant cells[44].

Their predominant concentration in malignantneoplasms leads to targeted local heating of tumours and the preservation of adjacent normal tissues under the influence of an alternating magnetic field. Despite the encouraging findings of preclinical trials of magnetic hyperthermia, there are several unanswered challenges in this area. This includes the establishment of optimal limits of magnetic field strength and frequency, their correlation with the duration of treatment, the toxicity of, magnetic nanoparticles (including the dependence of toxicity on the presence of specific ligands that improve the accumulation of magnetic particles in tumour cells), and determining their optimal concentration in the affected organ.

MRI and cancer diagnosis

There are several motivations for creating technology that can detect cancer in its earliest stages because early detection is linked to a favourable result with any sort of therapy[45]. Due to the availability of curative therapy, stage 1 cancer discovery is often linked with a 5-year survival rate higher than 90%[46,47]. Cancer may currently be found using a number of medical tests, including blood, urine, or imaging methods. When cancer is just a few millimetres (e.g., MRI) or centimetres (e.g., PET) in diameter, when it already contains more than a million cells, conventional imaging techniques often identify it. To address this drawback, molecular imaging has recently been proposed. Recent advancements in nanotechnology, molecular cell biology, and imaging technologies made it feasible to create this novel imaging modality. While magnetic resonance imaging (MRI), which offers the best spatial resolution when compared to other techniques and is noninvasive or at the very least minimally invasive, is of particular interest, molecular imaging imaging applies to a variety of imaging techniques such as Positron Emission Tomography (PET), computed tomography, or ultrasound.

Implementation of iron-based nanoparticles increased MRI sensitivity due to buildup of iron in the liver produced by selective action of the hepatobiliary system. Nanoparticles have been lately exploited by biologists, pharmacologists, physicists, doctors as well as pharmaceutical business. There are roughly 20 clinically approved nanomedicines utilized for the therapy. Some examples are Abraxane, an albumin-bound form of paclitaxel with cobalt of mean particle size of approximately 130nm that is used to treat breast cancer and Doxia, also based on Cobalt, is used for the treatment of refractory ovarian cancer and AIDS-related Kaposi’s sarcoma and it consist of nanoparticles with a polyethylene glycol coating. A fundamental aspect of nanoparticles delivery systems is their potential to boost transport systems is their ability to enhance the concentration of anticancer medicines in tumor cellssince certain nanoparticles passively accumulate in tumor's after their intravenous injection. Nanoparticles may pass via microscopic capillaries and are taken up by cells, which allow efficient drug accumulation at target locations and also sustained and regulated release of medications at target areas over a period of days and weak[45].

V. CONCLUSION

In this reviews article, the use of magnetic hyperthermia, targeted medication delivery, and nanoferrite in MRI was briefly discussed. Nanoferrites are a great choice for all of these biological applications due to their superb characteristics and adjustable magnetic behaviour. The main issues that need to be addressed when employing different nanoferrites for biomedical applications to increase effectiveness are stability and biocompatibility. Although there have been many in vitro research on nanoferrites, there haven't been many clinical trials on the substance.

VI. REFERENCES

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