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Research Article

Structural, Morphological and Magnetic Properties of Manganese-doped Copper Ferrite Nanoparticles

U. Naresh[1*], T. Ramprasad[2], Y. Saritha Kumari[3], S. Nagaveni[4]

[1]PVKK Institute of Technology, Jawaharlal Nehru Technological University, Anantapur, Andhra Pradesh
[2]Malla Reddy Institute of Technology and Science, Jawaharlal Nehru Technological University, Hyderabad, India
[3]Malla Reddy college of Engineering, Jawaharlal Nehru Technological University, Hyderabad, India
[4]CMR Institute of Technology, Jawaharlal Nehru Technological University, Hyderabad, India

**Corresponding Author: talluriramprasad123@gmail.com*

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Abstract: The current investigation focused on the structural, morphological, and magnetic properties of Manganese-doped copper ferrite nanoparticles ($Mn_{0.5}Cu_{0.5}Fe_2O_4$) (MCF) synthesised through the auto-combustion method. X-ray diffraction (XRD), Fourier transform infrared spectroscopy (FTIR), and field emission scanning electron microscopy (FESEM) are used to observe morphology, and the structural features, with the help of vibrational sample magnetometer (VSM) the magnetic characteristics were assessed based on VSM hysteresis behavior. The structural properties such as crystallite size and macro strain were evaluated using the Williamson-Hall method from X-ray diffraction analysis. The FTIR studies reflect the presence of octahedral and tetrahedral bond vibrations; this is evidence of the formation of spinel structure. Interestingly, the VSM studies represent the soft magnetic nature with lesser values of retentiveness and coercivity. The examined results reflect that the synthesized materials are most suitable for biomedical applications.

Keywords: Nano Ferrites, auto-combustion method: XRD, FTIR FESEM, And VSM.

1. Introduction

The Ferrites are part of ferromagnetic materials, meaning that they consist of iron oxide which is combined with other metal oxides typically those of zinc, manganese, nickel, magnesium, and other metals[1].They can be easily magnetizing and retain their magnetic properties after removing the magnetizing field. Their unique magnetic and electrical properties make them useful in various applications. The applications of ferrites depend on the type of ferrites, based on the hysteresis loop area the ferrites are classified into Hard(large),and soft ferrites (small), with high magnetic permeability of soft ferrites they are used in the applications of Transformers and inductors, High-frequency devices, and another one the Hard ferrites with a higher coercivity, it can be used to create permanent magnets for various industrial and consumer applications [2-3].However, the structure of the ferrites has a significant impact on their properties. Ferrites have a cubic spinel structure, general formula for ferrite is AFe_2O_4 (A= Cu, Mn, Co, Ni, Zn… etc.). The spinel structure is a specific arrangement of cubic close-packed octahedral and tetrahedral lattices of oxygen atoms and iron (Fe^{3+}) ions. However, the ferromagnetic nature is decided by the cations of octahedral sites [4]. The magnetic interactions between the iron ions in the octahedral sites are responsible for the strong magnetization. The magnetic and electrical characteristics vary depending on which metal cations are present in the tetrahedral and octahedral vacancies [5].Between all the ferrite substances the Copper ferrite ($CuFe_2O_4$) is an essential and valuable material with significant importance in various fields, especially in magnetic and electronic applications [6].However, the doping of different elements into copper ferrite ($CuFe_2O_4$) leads to changes in its crystal structure and it can significantly impact its physical properties like lattice parameters, the arrangement of cations in the crystal lattice, crystal symmetry, magnetic properties, electronic structure, phase transformation, and Microstructure. The effects, however, vary depending on the doping, its concentration, and the synthesis, all of which are important factors in defining the precise effects on the physical characteristics of copperferrite [7]. Many of the researchers focused on sorting the material's properties for specific applications, since; understanding the relationship between doping and structural properties is a crucial task to engineer a functional device with suitable materials with enhanced performance. For example, the Magnetic properties of Copper ferrite are suitable for magnetic recording similarly, its low magnetic losses at high frequencies meet the applications of Microwave devices, and its recyclability raised potential applications in the field of environmental and biodegradable applications

[8].The doping of copper ferrites with manganese can adjust their magnetic and electrical properties. The incorporation of Mn^{2+}ions leads to changes in the magnetic moment of the material. Depending on the concentration of Mn dopants, it is possible to tailor the magnetic properties, such as coercivity and saturation magnetization, making them suitable for different applications [9].Manganese (Mn) doped copper ferrites, often denoted as $Cu_{1-x}Mn_xFe_2O_4$, are a specific type of ferrite where some of the copper (Cu) cations in the crystal lattice are replaced by manganese (Mn) cations. This substitution introduces interesting properties which lead to potential applications in various fields, particularly magnetic and electronic devices. The Mn-doped copper ferrites have been extensively studied for various applications; the tunable magnetic properties of these ferrites make them suitable candidates for magnetic recording media, such as hard disk drives. The magnetic properties helped in the case of magnetic sensors for different applications, including position sensing and non-destructive testing [10].Because of their small magnetic losses at extremely high frequencies, Mn-doped copper ferrites are used in microwave devices like circulators and isolators. In addition, Mn-doped copper ferrites have also been explored for biomedical applications, especially in magnetic resonance imaging (MRI) and hyperthermia for cancer treatment steady agents.

Researchers continue to search the properties of Mn-doped copper ferrites to develop new strategies and applications in various fields, taking advantage of their unique magnetic and electronic properties. The magnetic properties of Mg-Cu-Co ferrites; for sample x = 0.03, their XRD data show that the lattice constant is 0.8370nm and the average crystallite size is 49.71 nm investigated Akhtar et.al, [10]. These observations confirm that dysprosium (x=0.03) doped for Mg-Cu-Co-soft ferrites may be applicable in transformer cores, microwave absorbance and telecommunication devices. The materials' resistivity was reduced by the effect of doping Mn on the produced La-doped copper ferrite in Mn-doped Tertiary $Cu_{0.25}$ $Ni_{0.15}$ $M_{0.25}$ $Co_{0.35}L_{0.15}$ $Fe_{1.85}$ O_4 composites synthesized by Aslamet.al [11].Sol-gel electro-spinning has been used to create novel manganese-substituted copper ferrite $Cu_{(1-x)}$ MnxFe$_2$O$_4$; (x= 0, 0.25, 0.5, and 0.75) nano fibers synthesized by Safartoobi et.al [12].Investigations were conducted into the fibres' optical, magnetic, elemental, structural, and morphological properties. The Williamson-Hall method results showed that the addition of Mn altered the average crystallite size and lattice strain of the samples. The FESEM images emphasized the formation of nano fibers increased by doping the Mn. composed of $Cu_xMn(_{1-x})$ Fe_2O_4 (X = 0, 0.05, 0.1, 0.15, and 0.2) ferrite nanoparticles by reverse co-precipitation, and the effects of varying percentages of Cu^{2+} doping on the structural and magnetic characteristics of manganese ferrite nanoparticles have been studied by Samani et al. [13].The produced nanoparticles' shape was found to be nearly uniform and spherical, while the estimated energy band UV-visible spectroscopic gap was found to decrease as Cu^{2+} levels increased. The sol-gel auto-combustion technique was used to create the nano-structured $Cu_{0.25}M_{0.75}$ Fe_2O_4 (M = Mn, Mg, Ni, and Co) ferrites. Their structural, magnetic, morphological, and optical characteristics were examined. There were no secondary phase peaks visible in the diffraction patterns, indicating that all the compounds were single-phased spirals according to the XRD study. Based on XRD measurements, Vegard's rule indicated that the average lattice constant of ferrites varied between 8.413 Å and 8.131 Å. Meanwhile, the higher annealing temperatures resulted in crystallite sizes ranging from 18.978 nm to 42.351 nm. Further analysis of the magnetic characteristics revealed that $Cu_{0.25}M_{0.75}Fe_2O_4$ (M = Mn, Mg, Ni, Co) ferrites' behavior changed from ferromagnetic to ferromagnetic as M was successively substituted, even though the Jahn-Teller effect greatly prevented this transition. Abinaya et.al [14] detected Cation occupancy of Mn, A in tetrahedral and B in octahedral sites using XPS, XRD, and VSM investigation. The Tauc plot of $Mn_xCo_{1-x}Fe_2O_4$ nanoparticles was used to estimate the energy band gap, and the results ranged from 1.123eVto 1.247eV. The occupation of Mn^{2+} and Mn^{3+} in octahedral and tetrahedral sites, respectively, is confirmed by XPS-based energy level splitting of Mn $2p_{3/2}$ and Mn $2p_{1/2}$As a result, several researchers published studies on the physical characteristics of Mn and Cu ferrite nanoparticles together. To examine the physical characteristics of the produced nanoparticles, we reported equivalent amounts of Mn and Cu-doped ferrite nanoparticles.

2. Materials and Method

Nano crystalline manganese-doped copper ferrite particles were created using an auto-combustion process. The chemical formula $Mn_{0.5}Cu_{0.5}Fe_2O_4$ was designed and stoichiometric calculations were carried out accordingly. The chemicals used were manganese nitrate (Mn $(NO_3)_2.6H_2O$), copper nitrate ($Cu(NO_3)_2.6H_2O$), and iron nitrate ($Fe(NO_3)_3.9H_2O$), which were dissolved in deionized water and combined with a magnetic stirrer. The solution was heated to 160°C until self-combustion occurred, and then it spontaneously cooled to room temperature. The pH level was continuously monitored during the procedure and significant gases were evolved. A dry, loose ferrite powder was created as a result. Several techniques were employed to characterize the structural, morphological, and magnetic properties of this tiny powder.

3. Results and Discussion

3.1. Structural properties

3.1.1 XRD analysis

The X-ray diffraction technique was used to characterize the spinel ferrite system $Mn_{0.5}$ $Cu_{0.5}Fe_2O_4$ structurally. Figure.1 shows the X-ray diffraction (XRD) pattern of the $Mn_{0.5}Cu_{0.5}Fe_2O_4$ ferrite system. The obtained pattern reflects the single-phase cubic spinel structure with reflected intensity peaks noted as (111), (220), (311), (222), (400), (422), (511), (440), (533), and (444) show that the materials were manufactured in a crystalline form.. The obtained spectra observed very intensive reflection planes and sharp and clear from the image there was no extra peak detected in the XRD pattern. The inter-planer distance and respective intensive reflection peak (311) were used to the find lattice constant from the formula

$$a = d_{hkl}/\left(h^2{}_{+}k^2{}_{+}l^2\right)^{1/2}\text{------------------}(1)$$

Where (**h k l**) is the Miller indices, '**a**' is the lattice constant, and'd' is the interplanar space. The lattice constant in this case was 8.6 A^0

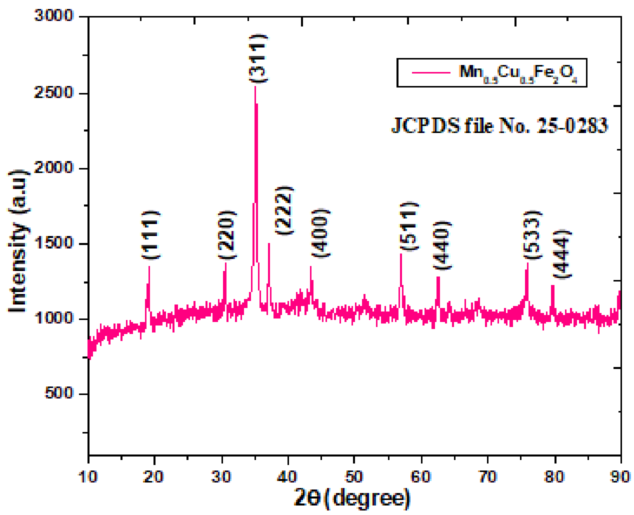

Figure.1. XRD spectra of $Mn_{0.5}Cu_{0.5}Fe_2O_4$Nanoparticles

The Average crystallite size(D), Dislocation density(ρ_d=(D)$^{-2}$),and Strain(ε=β/4tanθ),where β is full-width half maxima of XRD peaks of XRD spectra calculated. By applying the stranded Debye Scherrer equation D=0.9λ/βcosθ, the average crystalline size can be found to be 18.92 nm.

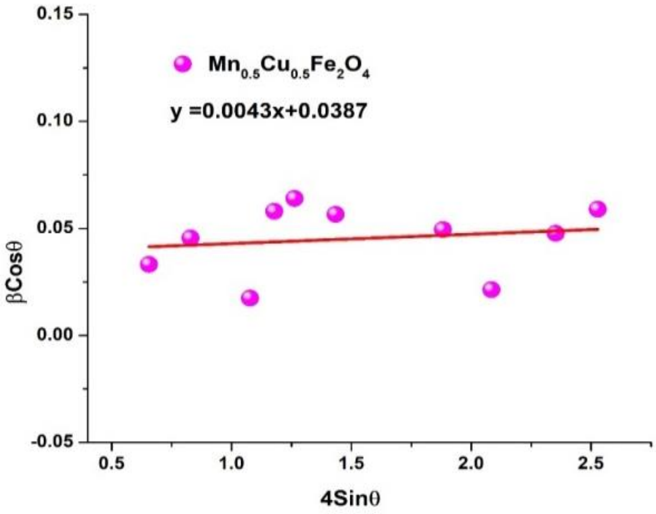

Figure .2 W-H plot of $Mn_{0.5}Cu_{0.5}Fe_2O_4$

T0he average crystallite size (D') and the micro-strain (ε') of the materials are determined by computing their mechanical properties using the W-H (Williamson-Hall) graph as shown in Fig.2. A linear fitting relation was used to create W-H graphs between βCosθ and 4Sinθ: βCosθ = 0.9λ/D' + ε' 4sinθ i.e. y = c + mx form), here the intercept value (D') is connected to the crystallite size, while the slope of the straight line (ε') is related to the micro-strain. Table 3.1 presents a tabulation of the derived values for ε' and D'. The table makes it clear that the values of ε' and D' agree with the values of ε and D that were determined using Scherer relations [15-16].

Table 3.1 Structural properties of $Mn_{0.5}Cu_{0.5}Fe_2O_4$

Structural properties	**$Mn_{0.5}CU_{0.5}Fe_2O_4$**
a (nm)	8.61
D(nm)	18.92015377
V nm^3	638.277381
ρ_t(g/cm^3)	0.52734918
ρ_e(g/cm^3)	0.45879378
porosity (P)	0.13
ε(x10^{-3})	2.9
D' (nm) W-H plot	19.1
ε' (x10^{-3}) W-H plot	3.12

3.1.2. FTIR analysis

The Fourier transform infrared spectrum (FTIR) of produced mixed phase and single phase of$Mn_{0.5}Cu_{0.5}Fe_2O_4$ (MCF) nanoparticles in the range of 2500–400cm^{-1} as shown in figure.3.The cations that are dispersed among the two sites (Octahedral and tetrahedral) can establish bonds with oxygen atoms, which may result in the creation of a spinal structure (AB_2O_4).

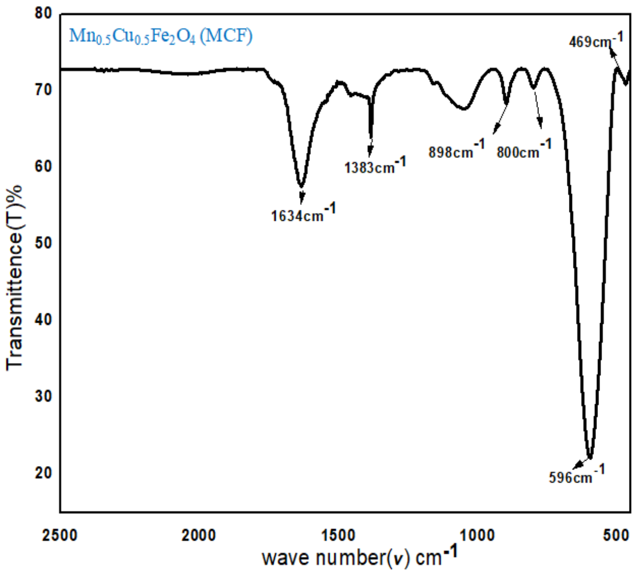

Figure.3.FTIR spectrum of $Mn_{0.5}Cu_{0.5}Fe_2O_4$ nanoparticles

In figure.3.the spectra peaks show two kinds of metal-oxide (MnCu-O& Fe-O) absorption bands, one is v_1 at 469 cm^{-1} and v_2 at 596cm^{-1}indicating tetrahedral spinal absorption band and the octahedral spinal absorption band conforms to the cubic spinal structure of MCF nanoparticles. From the spectrum, the other absorption peaks positions raised at 1634cm^{-1} and 1383cm^{-1} are present because of the O-H stretching and bending vibrations of H_2O molecules absorbed by MCF nanoparticles respectively [17]. Furthermore, Figure 3 suggested that the extra absorption bands surrounding the octahedral site might result from the synthesis-related production of ferrous ions. This resulted in standing evidence for the diffraction pattern in the cubic $MnCuFe_2O_4$ single-phase structure.

3.2. Morphological Properties

The morphological characteristics of $Mn_{0.5}Cu_{0.5}Fe_2O_4$ (MCF) nanoparticles were examined using FESEM Micrographs. The obtained FESEM photographs of $Mn_{0.5}Cu_{0.5}Fe_2O_4$ (MCF) nonmaterial are presented in Figure 4. It was observed that the micrograph consists of a cluster of particles. Because of the circumstances surrounding synthesizing, the grains in the fiber form were dispersed irregularly. Because of the circumstances surrounding synthesizing, the grains in the fiber form were dispersed in an irregular manner. But these materials have the ability to form clusters, which suggests they could be useful in biomedical applications.

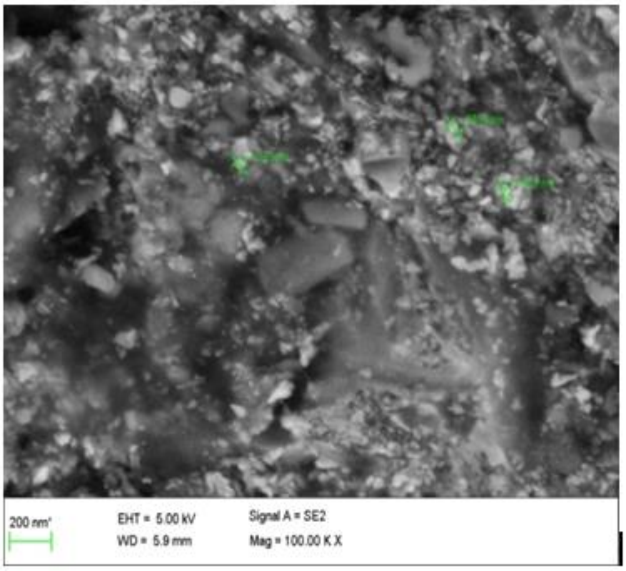

Figure. 4. FESEM image of $Mn_{0.5}Cu_{0.5}Fe_2O_4$

In addition, the linear intercept (LI) approach method was used to determine the average grain size (Ga) of the $Mn_{0.5}Cu_{0.5}Fe_2O_4$ (MCF) materials. By the LI method, average grain size(Ga) is given by=3d/2MN, where 'd' denotes the testing line distance, "M" denotes the applied magnification, and "N" denotes the number of grains in contact with the testing line. The grain size is determined to be 92 nm. Furthermore, the development of agglomerated nano fibers was ascribed to magnetic interactions between the nanoparticles. This morphology consists of elongated nanoparticles with enormous fibres that are encircled by nano spheres. Compared to particle size, grain size appeared to be greater. Normal explanations for it include the agglomeration effect among the grains or particles (produced due to high magnetic interactions). [18–19].

3.3. M-H Loop analysis or VSM studies

The study involved an analysis of the magnetic properties of copper ferrite nanoparticles which were doped with manganese. These nanoparticles were given the chemical name $Mn_{0.5}Cu_{0.5}Fe_2O_4$. For the analysis, a vibrating sample magnetometer (VSM) instrument was used. The magnetization (M) versus magnetic field (H) or M-H curve was studied to understand the magnetic nature of the material. This curve displays the hysteresis characteristics of the material. The M-H curve hysteresis loop was obtained at room temperature (300K) for the magnetic field range of -15KOe to +15KOe for the synthesized sample. The obtained results were presented in the form of Figure 5.

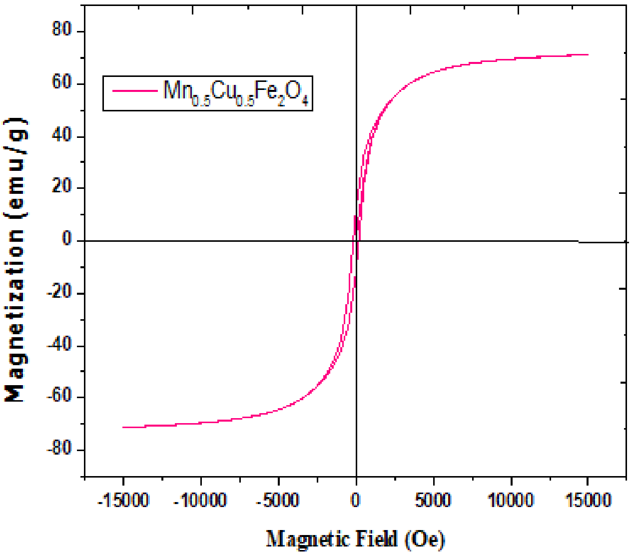

Figure.5. M–H loop Behavior of $Mn_{0.5}Cu_{0.5}Fe_2O_4$

The hysteresis of nano manganese copper ferrite (MCF) was analyzed to determine its magnetic properties. The saturation magnetization (Ms) was found to be 71.88 emu g^{-1}, while the remnant magnetization (Mr) was 11.44 emu g^{-1}. The Mr/Ms ratio was calculated to be 0.1591, and the coercive force (Hc) was measured to be 177.8 Oe. Additionally, the anisotropic energy K1 was determined to be 30087.5 erg cm^{-2}, which is consistent with recent findings that demonstrate exceptional magnetic characteristics [20-23]. Overall, the MCF nanoparticles exhibit a soft magnetic nature and are highly sensitive to magnetic behaviors.

4. Conclusion

The auto-combustion method was employed to synthesize manganese-doped copper ferrite nanoparticles, using a solution with an equal concentration. The synthesized nanoparticles were analyzed using X-ray diffraction (XRD), which confirmed that they have a cubic spinal structure. The sample was further analyzed, and its average crystalline dimension (D) and lattice parameter (a) were determined to be within the range of 18.62 nm and 8.61 nm, respectively. To examine the mechanical strain relation, W-H plots were employed. The sample was also analyzed using field-emission scanning electron microscopy (FESEM), which showed agglomerated stone-shaped grains. The grain size was calculated using the linear intercept (LI) approach, which revealed a size of 92nm. Fourier-transform infrared (FTIR) spectra results confirmed the cubic spinal structure of MCF nanoparticles. The synthesized nanoparticles were further analyzed using vibrating sample magnetometer (VSM) photographs, which revealed that they exhibit a soft-magnetic nature. These materials have a future scope in the field of biomedical applications.

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Conflict of Interest

This copy has not been communicated or is in process anywhere else. Therefore, there is no conflict of the addicted for us to encounter.

Data Availability

The authors are ready to communicate any data regarding this research in further information.

Funding Resources

Funding Source There was no external funding for this study.

Authors contribution

The first author Dr Naresh synthesized samples and analyzed the structural and magnetic properties of samples. The author T Ramprasad helped to calculatestrain-related W-H analysis and other authors helped much during the characterization and writing of the papers effectively.

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AUTHOR'S PROFILE.

Dr .U. Naresh is currently working as assistant professor in PVKK Institute of Technology, Anantapur Andhra Pradesh, India. He published 15 research papers in national internation journals in the field of magnetic ferrites. He contributed 5 book chapters related to polymers and graphene-based materials.

Dr.T. Ram Prasad earned his B. Sc and M. Sc in Physics from Acharya Nagarjuna University in 2004 & 2007 respectively. He accomplished a Doctorate in Physics from Rayalaseema University, Kurnool, Andhra Pradesh in 2019. He is currently working as Associate Professor and Head of the Department of Basic Sciences and Humanities in MRITS, Hyderabad since 2019. He published in 12National and International journals, and conducted workshops and FDP's in Physics. His main research work was focused on Material Science in Physics. He has 16 years of experience driven to inspire students to pursue academic and personal Excellence.

Y. Saritha Kumari earned her B.Sc from Kakatiya University in 1999-2002, M.Sc in Physics from Acharya Nagarjuna University in 2002 & 2004 And M.Phil in Material Science from Acharya Nagarjuna University in 2006 & 2008 respectively. She submitted Ph.D. Thesis in Material Sciences, GITAM (Deemed) University, Hyderabad, Telangana in 2019 to 2023. She is currently working as Associate Professor in Physics, MRCE, and Hyderabad since 2020. She published in various National and International journals, and Attended workshops, Conferences and FDP's in Physics. Her main research work was focused on Material Science in Physics. She has 20 years of experience driven to inspire students to pursue academic and personal Excellence.

Dr. S. Nagaveni earned her B. Sc from Kakatiya University and M. Sc in Physics from Acharya Nagarjuna University in 2003 & 2006 respectively. She accomplished a Doctorate in Physics from Jawaharlal Nehru Technological University, Hyderabad, Telangana in 2017.She is currently working as Associate Professor in Physics from Freshmen Engineering Department, CMRIT, Hyderabad since 2023. She published in various National and International journals, and conducted workshops and FDP's in Physics. Her main research work was focused on Biophysics Specialization in Physics. She has 17 years of experience driven to inspire students to pursue academic and personal Excellence.