Synthesis and Characterization of CuFe2O4 Spinel Ferrite Nanoparticles by One Step Sol-Gel Auto-combustion Method

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Abstract:
In the field of nanomedicines spinel ferrites have gained more attention for their various biomedical applications. The metal spinel ferrites have been used for drug targeting, cancer therapy, medical imaging, diagnostic tool, antibacterial agents and many more. The smaller particle size, biocompatibility, magnetic properties, chemical and thermal stabilities have made them popular for future research. The copper spinel ferrite is one of the spinel ferrites has gained more attention for their unique antimicrobial activity and drug targeting. The development of copper ferrite nanoparticles involves many hazardous chemicals and need multiple steps. In this study we prepared copper ferrite nanoparticles by one step auto combustion method and characterized for future application.
Keywords: Spinel Ferrite; Nanoparticles; Auto combustion method; Characterization.
How to cite this article: Gautam BS, Sahu SK, Jana U. Synthesis and Characterization of CuFe2O4 Spinel Ferrite Nanoparticles by One Step Sol-Gel Auto-combustion Method. Int J Drug Deliv Technol. 2026;16(10s): 657-662;
DOI: 10.25258/ijddt.16.10s.79

1. Introduction

Spinel ferrites nanoparticles have recently been investigated for their biomedical applications, especially for drug delivery systems. Their biocompatibility, antibacterial activity, magnetic properties, chemical and thermal stabilities have made them popular for future research as evidenced from various scientific literatures (Ansari et al., 2017; Kefeni et al., 2020). The common structure of metal spinel ferrite is MFe_2O_4, where, M stands for the metallic cations of Cu, Ni, Co, Zn etc (Ahmad et al., 2016). Metallic nanoparticles have been used for targeted drug delivery systems, to kill tumor cells. The other medical applications reported are magnetic resonance imaging (MRI) (Yang et al., 2015), biosensors, detection of proteins, carbohydrates, and destruction of viruses and bacteria (Ghosh et al., 2021a; Jermy et al., 2022; Joudeh and Linke, 2022). Numerous recent experimental findings have demonstrated the significance of metal spinel ferrite nanoparticles in cancer therapy, antibacterial activity and diagnosis of diseases (Aisida et al., 2020; Kefeni et al., 2020; Spirou et al., 2018). However, none of the metal spinel ferrites have received approval for biomedical applications. The primary issue appears to be the absence of data regarding their potential toxicological implications for both short and long-term exposure.

Nowadays, focus has been given to develop biocompatible nanoparticles for drug targeting and disease treatment. Nanomaterials exhibit distinct physical and chemical properties, enhancing their efficiency in targeting therapeutic agents to specific sites while minimizing side effects. The small size, high surface-to-volume ratio, distinctive structural properties, and enhanced ability to penetrate cell membranes of nanomaterials contribute to their effectiveness in interacting with microbial membranes, demonstrating prolonged antimicrobial activity (Ghosh et al., 2024, 2021b). The targeting capability of nanoparticles renders them advantageous as novel drug carriers. Consequently, the development of an appropriate biocompatible nanocarrier for drug delivery has the potential to revolutionize biomedical research.

$CuFe_2O_4$ has become promising materials for developing new antimicrobial agents and drug carriers. The smaller particle size of $CuFe_2O_4$ carrier have high cell permeability and good saturation magnetization (Sun et al., 2008). They also exhibit electrical properties with good chemical and thermal stabilities (Rashad et al., 2012). $CuFe_2O_4$ is used in hyperthermia treatment and it has good intrinsic loss power (ILP) value which is suitable for the hyperthermia therapy. It is also a better approach than conventional magnetic nano-carrier therapy(Fotukian et al., 2019). Various methods have been reported for the preparation of spinel ferrite nanocarriers, including co-precipitation, electrochemical processes, thermal decomposition, and electrospinning techniques (Fantechi et al., 2015; Kefeni et al., 2020; Mazario et al., 2012; Zi et al., 2009). The sol-gel auto combustion method is a widely utilized technique for synthesizing spinel ferrite nanoparticles due to its simplicity, cost-effectiveness, and rapid execution (Ghosh et al., 2024; Sun et al., 2008, 2007). Considering the potential applications of spinel ferrite, significant focus has been shifted to synthesize copper spinel ferrite nanoparticles by simple one step method. In this work a simple auto combustion method was adopted to prepare the copper spinel ferrite nanoparticles. The nanoparticles were characterized further for their suitability.

2. Materials and Methods

2.1 Materials

Cupric nitrite [$Cu (No_3)_2\ 3H_2O$] and ferric nitrate [$Fe (NO_3)_3,\ 9H_2O$] were purchased from Sigma Aldrich, Mumbai. Anhydrous citric acid was purchased from Loba Chemicals, Mumbai. Deionized water was purchased from Aagam Chemicals, Nagpur, India. All other chemicals used were analytical grades.

2.2 Synthesis of copper ferrite nanoparticles

Copper ferrite nanoparticles were prepared by the previously reported sol-gel auto-combustion method with slight modifications (Naseri et al., 2013). Approximately 1 gm of cupric nitrite and 2 gm of ferric nitrate were placed in a beaker containing 50 ml of deionized water. Both the salts were mixed with continuous stirring by means of a magnetic stirrer for 2h. About 1 gm of anhydrous citric acid was added to the solution. The mixture was then stirred at constant temperature of 120 ^{o}C to get soft gel. The product was then poured into a petri dish and heated at 100 ^{o}C temperature for 24h to evaporate the water. The dried copper ferrite nanoparticles were cooled, and crushed into powder and stored in a well closed container for further evaluation.

2.3 Characterization of copper ferrite nanoparticles

The structure of copper ferrite nanoparticles was evaluated by XRD analysis using X-ray diffractometer (Shimadzu 6000, Japan) with a Cu Kα line as the source of radiation. The powdered sample was scanned to obtain diffraction pattern at a temperature in 2θ range of 10 to 80^{o}. The characteristic peaks of copper ferrite nanoparticles were obtained by Fourier transform infrared spectroscopy (Spectrum two FT- IR spectrometer, PerkinElmer, USA). The synthesized copper ferrites nanoparticles were scanned in the range of 4000-400 cm^{-1}. The recorded spectra were then analyzed for functional groups and spinel ferrite formation. The thermal behavior of copper ferrite nanoparticles was also conducted by Differential Scanning Calorimetry (DSC) (NETZSCH DSC 404 F3 Pegasus). The microstructures of copper ferrite nanoparticles were studied using scanning electron microscope (ZESS EVO 18, Germany) coupled with EDAX for elemental analysis.

3. Results and discussion

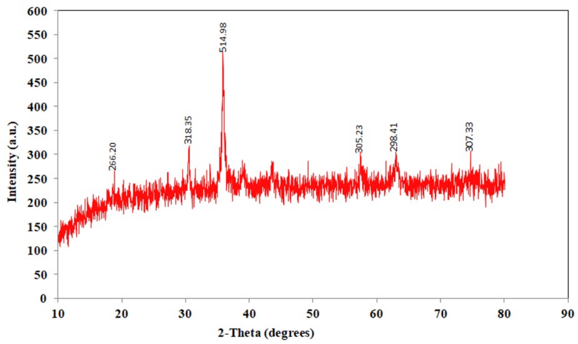
3.1 Synthesis of copper ferrite nanoparticles

Copper ferrite nanoparticles were synthesized by the sol-gel auto combustion method (Fig. 1). The sol gel auto combustion method is simple, quick, easy method for production of nanoparticles. The citric acid was used as a fuel for the mixture of cupric nitrite and ferric nitrate. Deionized water was used as a solvent in the auto combustion method for the formation of copper ferrite nano materials. The pH of the mixture was fixed at 7. The formation of ferrite nanoparticles occurs gradually during heat treatment. Additionally, it was found that the drying process had a significant impact on the development of nanoparticles since smaller particles had higher energy levels on their surface, which caused Ostwald ripening to produce larger particles (Ansari et al., 2017; Qu et al., 2006).

Fig.1. Synthesis of copper ferrite nanoparticles

3.2 Characterization of copper ferrite nanoparticles

3.2.1 X-ray diffraction (XRD)



The XRD analysis was carried out based on brags law to study the crystalline phases and crystal structures of materials. It helps to determine the arrangement of atoms and physical properties of materials. The high intensity peaks in the spectrum confirmed the crystalline nature of the synthesized ferrite nanoparticles. The XRD spectrum of $CuFe_2O_4$ is shown in Fig.2. The spectrum showed the different peak rang at 266, 318, 514, 305, 298, and 307. The highest intense peak 514 was observed at $2\theta = 35.86^0$.

Fig.2. The XRD spectrum of copper ferrite ($CuFe_2O_4$) nanoparticles

3.2.2 Fourier Transform infrared spectroscopy (FTIR)

FT-IR spectroscopy serves as a valuable technique for the analysis of multi-component systems, offering insights into both the blend composition and the interactions between polymers. Fourier transform infrared spectroscopy is a form of vibrational spectroscopy and technique used to identify organic, polymeric and in some cases, inorganic materials**.** The synthesized copper ferrite nanomaterial samples were scanned in the range of 4000-400 cm^{-1} (Fig.3). FTIR spectroscopy analyzes the functional groups, chemical bonds of the sample. The spinel ferrite formation is confirmed by the Fe-O vibrational modes, hydroxyl and other water peaks indicate surface-absorbed moisture. The peak ranges between 539.77 cm^{-1} and 480.62 cm^{-1} is due to Fe-O and Cu-O stretching vibrations, confirms the formation of $CuFe_2O_4$ structure, where Fe and Cu are present in tetrahedral and octahedral sites. The peaks between the wave number 3748 cm^{-1} and 2988 cm^{-1} indicate the O-H stretching vibration due to absorbed water or hydroxyl group. The peak ranges at 1340 cm^{-1} and 1048 cm^{-1} confirm the C-O stretching. Peaks between 600 to 700 cm^{-1} is due to metal oxygen stretching (Fotukian et al., 2019). The FT-IR spectra of $CuFe_2O_4$ nanoparticles (Fig.3) clearly demonstrate the absence of any residual organic compounds, thereby confirming the synthesis of organic-free copper ferrite nanoparticles.

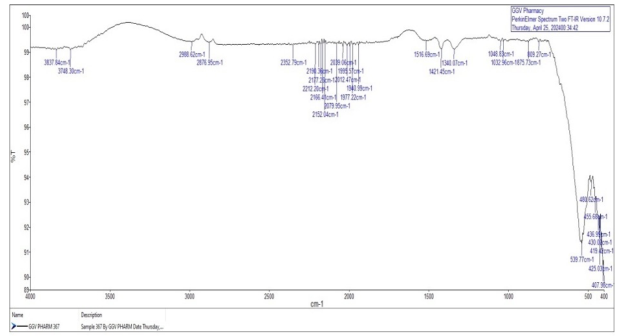


Fig.3. FTIR spectra of copper ferrite ($CuFe_2O_4$) nanoparticles

3.2.3 Differential Scanning calorimetry (DSC)

Differential Scanning Calorimetry is a thermal analytical technique used to study the thermal behavior of nanomaterials, polymers, pharmaceuticals and determine their crystallization temperature, glass transition temperature, melting point and thermal stability. The exothermic peak observed at 230-260 °C in the DSC thermogram results from the production of the copper ferrite spinel phase. This occurs when two oxides react with one another in a solid-state process to produce spinel ferrite (Dave, 2022). The lack of further peaks signified the nanoparticles' purity. The sharp peak results from the crystallization of metal ferrite, as corroborated by XRD analysis.

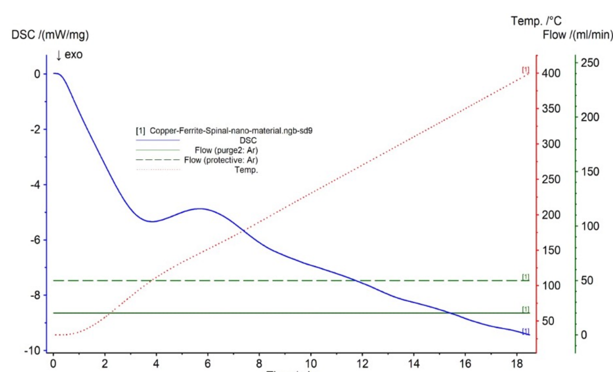


Fig.4. DSC thermogram of copper ferrite $CuFe_2O_4$ nanoparticles

3.2.4 Scanning electron microscope (SEM)

The scanning electron microscopy (SEM) was carried out for the synthesized $CuFe_2O_4$ nanoparticles to analyze the morphology, shape and size by scanning the surface with focused fine beam of electron. The SEM image confirms the size of nano-carrier which is in nano range.

The particle size is less than 200 nm with their spherical shape (Fig.5). The average particle sizes ranged from 58 nm to 107nm. Particles are spherical in shape and fairly uniform potential for high surface area and stability. The presence of gap in the image is due to the release of gas (carbon dioxide) during combustion process.

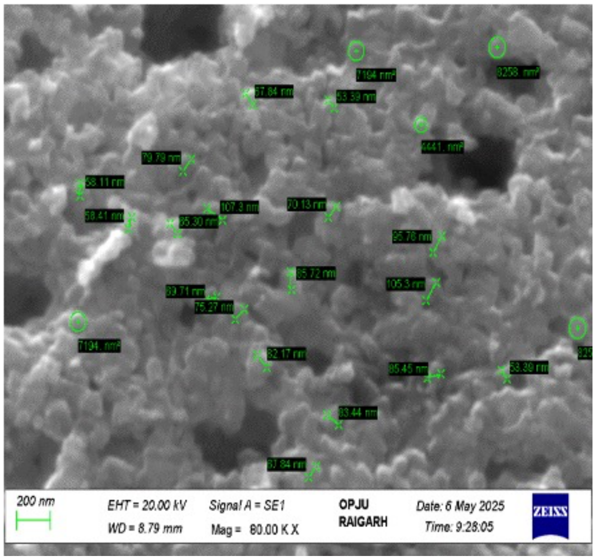


Fig.5. SEM image of copper ferrite ($CuFe_2O_4$) nanoparticles

3.2.5 Energy dispersive analysis of X-rays (EDAX)

The compositional analysis was studied by SEM equipped with oxford-energy dispersive X-rays system. EDAX is strong tool for micro-chemical characterization of materials with in a small volume of around 2µm depth.

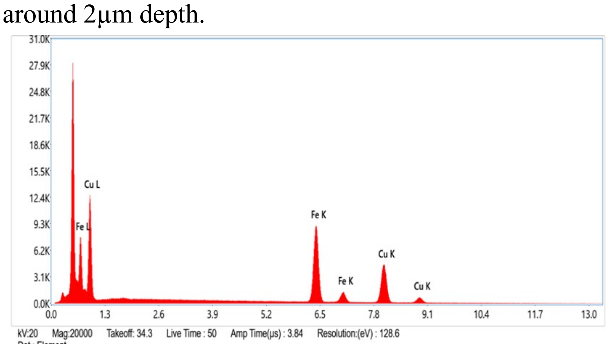
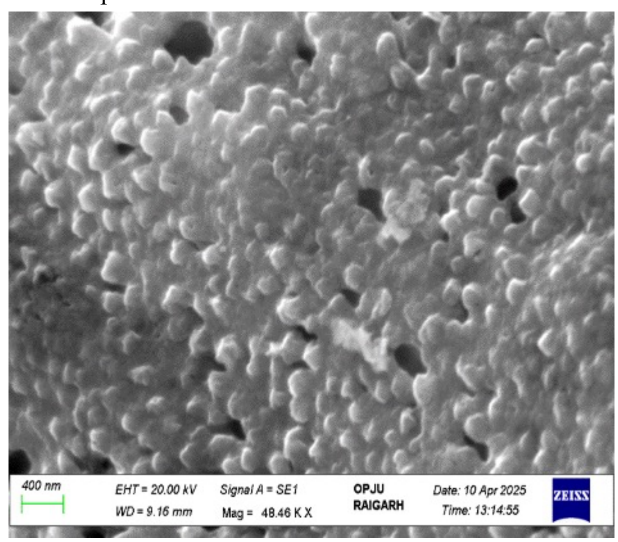


Fig.6. EDAX of copper ferrite ($CuFe_2O_4$) nanoparticles

Energy dispersive analysis of X-rays is used to determine the elemental composition of the sample ($CuFe_2O_4$). Peaks in the spectrum correspond to the energies of X-rays emitted by element present in the material (Atia et al., 2016). EDAX analysis revealed the presence of copper and iron in the sample (Fig.6) which is related to $CuFe_2O_4$ nanoparticles.

4. Conclusion

In this study we focus on the development of copper spinel ferrite nanoparticles by one step auto combustion method. The nanoparticles were successfully prepared. The crystal structure of copper ferrite particles was confirmed by XRD analysis. The sharp and intense peak in the spectrum indicating a well crystallized phase. The FTIR study confirmed oxygen metal bond at the octahedral sites and tetrahedral sites. The SEM analysis confirmed the nano size of the materials with spherical shape. The EDAX graph confirmed the presence of copper and iron which proves the formation copper ferrite compound. The DSC curve confirmed the exothermic transition around 200-260 ºC indicated the thermal stability of the synthesized spinel ferrite structure. The experimental results presented in this study are encouraging for future applications, particularly in biological disciplines. Additional research is currently underway to examine the drug delivery potential of copper spinel ferrite nanoparticles for multiple diseases.



Conflicts of interest: There are no conflicts to declare.

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