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Synthesis and characterization of Cadmium Substituted Copper Nano-Ferrites

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Abstract: This report presents the synthesis of copper cadmium nano-ferrite ($Cu_{1-x}Cd_xFe_2O_4$), x=0.3, 0.4, 0.5, 0.6 and 0.7) by Sol gel method and its subsequent characterization by using X-ray diffraction (XRD). Cadmium substituted copper nano-ferrites were synthesized using the sol-gel autocombustion method, which offers advantages such as low cost, simplicity, and the ability to produce nanoparticles with controlled size and morphology. Copper cadmium nano ferrites

(Cu-CdFeO₄) have emerged as promising materials due to their unique structural, magnetic, and electrical properties, making them suitable for diverse applications in electronics, catalysis, and biomedical fields.

Keywords: XRD, Combustion Method, Structural Properties, $Cu_{1,x}Cd_xFe_2O_4$.

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Introduction:

Spinel ferrites have been studied extensively due to ease to synthesis and abundant uses in technological and industrial applications. The useful properties of the spinel ferrites mostly depend upon the chemical composition, preparation methods, sintering temperature, nature of the additives and their distribution as reported earlier by Kefeni et al., (2020).

Among the spinel structures, Cu-Cd nano-ferrite has been widely used in different kinds of materials. Thus, the magnetic and electric properties of nickel ferrite have been researched and improved. Cu-containing ferrites form an interesting group of ferrites because of their typical electrical and magnetic properties and change in crystal structure on thermal treatment. Cu-Cd nanoferrites are low cost materials and have important magnetic and electrical properties for technological applications in Magnetic device, such as inductors, magnetic heads, magnetic refrigeration and magnetic

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resonance imaging. Nano ferrites have gained significant attention in recent years due to their unique magnetic, electrical, and structural properties, which make them suitable for various technological applications, including magnetic storage, sensors, catalysis, and biomedical uses. Among them, spinel ferrites MFe $_2$ O $_4$, (where M is a divalent metal ion) have been widely studied due to their tunable magnetic and electrical characteristics as reported earlier by Sayed, (2009).

The incorporation of different metal ions into ferrite structures allows for the modification of their properties, making them adaptable for specific applications.

Copper cadmium ferrites ($Cu_{1-x}Cd_xFe_2 O_4$) represent an interesting class of spinel ferrites in which the substitution of Cd^2 ions influences the material's structural, electrical, and magnetic behavior. Copper ferrite ($CuFe_2 O_4$) typically exhibits mixed spinel characteristics with both normal and inverse spinel phases, contributing to its unique electrical and magnetic properties. The inclusion of cadmium (Cd^2) alters the cation distribution between tetrahedral and octahedral sites, significantly affecting magnetization, coercivity, and dielectric properties. Understanding these effects is crucial for optimizing Cu-Cd nano ferrites for applications such as high-frequency electronics, electromagnetic interference (EMI) shielding, and microwave devices as earlier reported in by Natrajan , (2011).

In this study, Cu-Cd nano ferrites were synthesized using sol-gel, and their structural, and electrical properties were systematically analyzed. The impact of cadmium substitution on the crystallographic structure, grain size, saturation magnetization, and electrical conductivity was investigated to explore their potential applications. The results of this study provide insights into the role of Cd²⁺ ions in modifying ferrite properties, paving the way for the development of optimized nanomaterials for advanced technological applications as Reported in (Roumaiah K., 2008).

Materials and Methods:

The process for synthesizing copper cadmium ferrites begins with the preparation of nitrate solutions

according to Thirugnanam and gaya (2011) Individual solutions of copper nitrate, cadmium nitrate, and ferric nitrate were dissolved in deionized water, maintaining a stoichiometric ratio of Cu: Cd: Fe as 1:1:2 or according to the desired ferrite composition.

These metal nitrate solutions were then mixed in a beaker. Next, citric acid was added to the mixed solution as a chelating agent, with a molar ratio of citric acid to the total metal cations set at 4:1:1:2. The solution is stirred vigorously to ensure it becomes homogeneous as explained by Mahmoud and Hassan, (2016).

Following this, the pH of the solution was adjusted to around 6-7 by slowly adding ammonium solution or NaOH dropwise. This promotes proper complexation of the metal ions, aiding in the formation of the gel. The solution was then gently heated to around 70-80°C on a hot plate with continuous stirring. This gradual heating removes excess water and leads to the formation of a viscous gel. Optionally, a small amount of ethylene glycol can be added as a stabilizer to prevent the gel from drying too quickly and cracking as explained in by Dash AK, (2022).

Once the gel was formed, it was transferred to an oven and dried at approximately 100°C for several hours (typically 6-12 hours). This drying process removes any remaining water and initiates the polymerization of the gel. The dried gel transforms into a porous, fluffy solid known as xerogel. The xerogel was then subjected to combustion, where it undergoes self-sustained ignition, resulting in the formation of a fine powder. This occurs due to the decomposition of nitrates and the oxidation of citric acid, releasing energy.

After combustion, the resulting powder was calcinated in a muffle furnace at temperatures between 500°C and 800°C for about 2-4 hours. This Calcination step enhances the crystallinity of the copper cadmium ferrite and promotes phase formation. The calcinated product is then cooled to room temperature and ground using a mortar and pestle to ensure a uniform particle size, (Fig.1) As shown in (Verma K, 2011).

Gimpses to stem performed to synthesis copper cadmium nano-ferrite:





Fig. 1. Preparation of Sample

Results and Discussion:

In this work, the structural properties of Cu-CdFe₂O₄ and Crystalline size were analyzed. The X-ray diffractograms of CuCdFe₂O₄ ferrite system (Fig. 1) reveal the single-phase spinel structure without any ambiguous reflection. The experimentally observed d spacing values and relative intensities are in well agreement with those reported in the ASTM powder diffraction files. The calcined powder samples shows XRD peaks at D = 79.512651 nm and 80.571207 nm, (Fig. 2) In general, copper ferrites are inverse spinel ferromagnetic materials. However, the degree of inversion is deeply related to the synthesis techniques adopted. However, just like zinc, cadmium has a strong tetrahedral preference which makes the a-b interaction effect the net magnetic moment in AB,0, ferrite. However, a higher magnetic moment can be achieved if the cadmium ions can move to the octahedral sites.

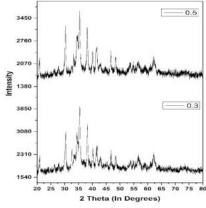


Fig. 2. XRD Pattern of Nanoparticles from Cadmium Substituted copper Nano Ferrite

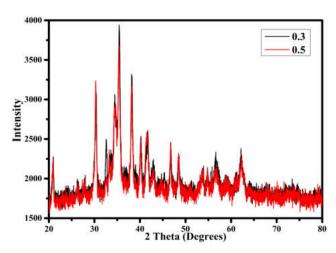


Fig. 3. Comparison of XRD Pattern at 0.3 & 0.5 Concentration

The size of the nanoparticles is calculated by using Scherrer Equation.

Scherrer equation relates the size of sub micrometer crystallites in solid to broadening of peaks in a diffraction pattern. It is used in the determination of size of the crystals in powder form.

Mathematically, Scherrer equation can be written as:-

$$D = \frac{\kappa \lambda}{\beta \cos \Theta}$$

Where,

D is the mean size of the order domain (crystalline size)

K is dimension less shape factor (Scherrer's constant)

 λ is wavelength of X ray

B is width at half of maximum intensity

 θ is Bragg's angle

FOR 0.3 MOLE

The Miller indices of the peaks are:-

$$2\theta = 30.28$$

$$\theta = 15.14$$

 $\beta = 0.0178$

K = 0.9

 $\lambda = 1.518$

After substituting the values in the formula, we have obtained the value of D is:-

D=0.9*1.518/0.0178*cos(16.15005)

= 79.512651 nm

For 0.5 MOLE

The miller indices of the peaks are:-

$$2\theta = 35.42$$

$$\theta = 17.71$$

$$\beta = 0.0178$$

$$K = 0.9$$

$$\lambda = 1.518$$

After substituting the values in the formula, we have obtained the value of D was obtained:

$$D = 0.9*1.518/0.0178*cos(17.71)$$

 $= 80.571207 \, \text{nm}$

Hence, the nanoparticles of copper cadmium nanoferrites by our group are in the range of nanoscale, having the diameter of 79.512651 nm and 80.571207 nm.

Conclusion:

The synthesis was successfully done according to the used methodology i.e. Sol gel synthesis.

The prepared solution was of expected colour i.e. brownish black.

It was compliance with the verified XRD experimental result, the prepared copper cadmium nano-ferrite nanoparticles was crystalline in nature which was indicated by sharp peaks of XRD graph (Fig. 3.).

The calculated size of prepared nanoparticles is in nano range having diameter of

D = 79.512651 nm and 80.571207 nm.

According to literature survey, the structural properties and catalytic activity of copper cadmium nano-ferrites have been studied till now. The valuable outcomes of the study, motivates the researchers to further investigate various other properties of the materials and their applications. The future work can be summarized as:

 These materials can also be synthesized via various other methods like: co-precipitation method, hydrothermal method and laser ablation method. Beyond the structural and catalytic activities, other properties such as electrical, dielectric and optical properties of the synthesized materials can be studied.

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