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# Crystallographic Nature And Textural Analysis On Copper Ferrites By Auto-Combustion Technique

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***Abstract:*** In this research work, Copper Ferrites ($CuFe_2O_4$) nanocrystalline powder with different calcination are synthesized from copper nitrate hexahydrate, Ferric Nitrate, Citric acid and chelating agent Ammonium hydroxide chemical precursors. Copper Ferrites nanocrystalline powder were fabricated by using sol-gel auto combustion technique. Crystallographic information, d-spacing and average crystallite size were characterized by X-Ray diffraction (XRD). The surface morphology and textural studies were examined by Scanning Electron Microscopy. The vibrational and functional group analysis of synthesized $CuFe_2O_4$ nanocrystalline powder by Fourier Transform mid-range Spectroscopy. The characteristics of synthesized $CuFe_2O_4$ nanocrystalline powder under above analysis information are economical and quite feasible for secondary electrode in Solid oxide fuel cells (SOFC) research field.

***Keywords*** **– Copper Ferrites, Auto-combustion, Calcination, XRD, SEM, FTIR.**

## I. INTRODUCTION

Ferrites are ceramic materials made from iron oxides with chemical addition of one or more metals. Ferrites are the compounds of two metallic oxides consisting iron oxide and any of bivalent elements (Ni, Mn, Mg, Zn, Cu, Fe, etc.). Ferrites show evidence of excellent electrical and morphological properties. Ferrites are dark grey or black, hard and brittle ceramic materials [1]. Copper spinal ferrites were prepared by self-high-synthesis (HTS), polymer matrix precipitation, citrate composition, coprecipitation, hydrothermal, crystallization, low solid-state reaction, sol–gel processing, microwave synthesis [2]. The structure properties and the confirmation of cubic inverse spinel phase of copper ferrite nanoparticles are studied in the present work using the X-ray diffraction (XRD). The investigation of surface morphology of ferrite samples was carried out by using scanning electron microscope (SEM) [3]. Ferrites have also been used in wastewater treatment processes, carbon dioxide decomposition for the utilization of carbon as solar hydrogen carriers, hybridization process for mixing solar and fossil fuel sources, and conversion of solar energy into hydrogen energy [4]. In addition, ferrites are simple to synthesize, have low cost reproducible and as well when compared to other gas sensors have structural and compositional flexibility and hence place them in a better place in the field of gas sensor technology [5].

## II. EXPERIMENTAL PROCEDURE

Initially, 1M of iron nitrate precursor solution are thoroughly mixed with digital magnetic stirrer with under constant stirring with 400 rpm at ambient temperature for half an hour. After that, 0.3 M of copper nitrate hexahydrate and the combination of 0.18 M of chelating agent citric acid are mixed with de-ionized water. The iron nitrate solution is then stirred using a stirrer for approximately 30 min under controlled conditions. Throughout this stirring process, the temperature is carefully maintained within the range of 80 to 100 °C. Subsequently, the copper nitrate solution is added drop by drop, followed by the gradual addition of citric acid in the same manner. Citric acid serves as the purpose of adjusting the pH level to (5~ 7), which is confirmed by testing with pH indication paper. After a few hours, the solution undergoes a phase transition, forming a Xerogel state. Finally, auto-combustion process has been occurred, resulting in the formation of copper ferrite with puffy state. By the conclusion, the observable copper ferrites powder is calcined with different temperature ranging from 500°C to 600°C for 2 hours respectively. The step-by-step procedure was depicted in Fig (A and B).


**Fig.1**. Flow chart of the step-by-step preparation and characterization of as-prepared $CuFe_2O_4$ powde

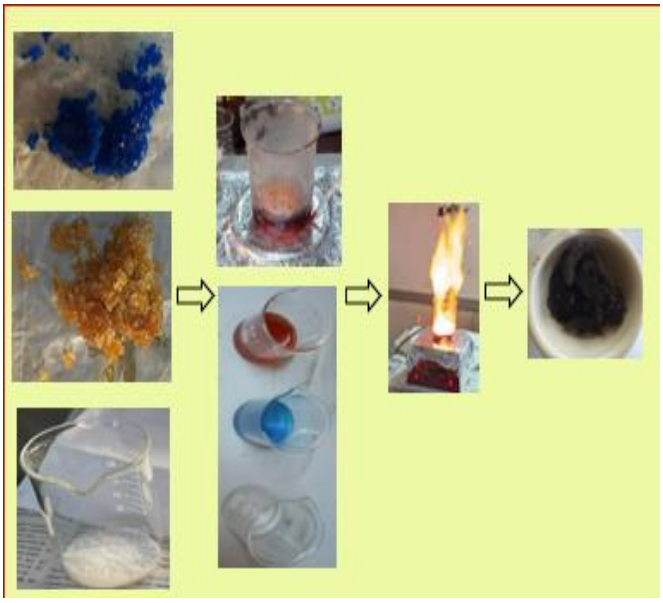

**Fig.2.** Photographic procedure for synthesized $CuFe_2O_4$ powder

## III. RESULTS AND DISCUSSION

### *Characterization*

The X-ray diffraction patterns of the sample was identified using X-ray Diffractometer (XRD-6000 from SHIMADZU) using CuKα radiation (λ = 1.5406 Å), using (2θ) =10°- 80° in steps of 5deg/s. The surface morphology was analysed through scanning electron microscope (SEM) with COXEM30$^+$ (0.5- 30) KV type. The Fourier transform infrared (FT-IR) spectra of the powders were recorded using FTIR spectrometer (FTIR 8400S from Shimadzu) in the wave number range (400-4000 cm $^{-1}$).

### 3.1 XRD characterization

XRD pattern and of the $CuFe_2O_4$ nanoparticle calcined at (500 and 600) °C for 3 h. The XRD patterns confirmed that of the sample is converted to the required ferrite. The value calculated experimentally of d-spacing and relative intensities of all reflection peak is fully compatible with the standard ICCD Card no (00-025-0283) for copper ferrite, respectively. For of the sample prepared and calcinating at different temperatures, it was noticed that when the temperature increased, the peak of the reflections became sharp. This indicates an improvement in the crystal when the calcinating temperature increases. Additionally, it is noticed that as the calcinating temperature increases, the width of the central maxima decreases. This is due to the grain size increase with an increase in the calcination temperature. The crystallite size (D) has been calculated using the high intensity peak for the composition using Scherrer's formula. The XRD observable analysis data exhibited at following Table [A] and [B].

Debye Scherrer's Formula;

$$D = 0.9\,\lambda / \beta \cos\theta \qquad (1)$$

Table 3.1 Observable crystallite size of synthesized copper ferrites calcined at 500°C

| Sr No | XRD analysis data for $CuFe_2O_4$(500°C) | | |
|---|---|---|---|
| | 2 Theta (θ) | Miller Indices | D (nm) |
| 1 | 35.6367 | 3 1 1 | 10 |
| 2 | 37.2783 | 2 2 2 | 7.46 |
| 3 | 62.9217 | 4 4 0 | 6.71 |
| 4 | 79.4664 | 5 1 1 | 5.85 |
| 5 | 74.4585 | 5 3 3 | 6.57 |
| 6 | 30.2538 | 2 2 0 | 9.29 |
| | | Average | 7.7 nm |

Table 3.2 Observable crystallite size of synthesized copper ferrites at calcined 600°C

| Sr No | XRD analysis data for $CuFe_2O_4$(600°C) | | |
|---|---|---|---|
| | 2 Theta (θ) | Miller Indices | D (nm) |
| 1 | 35.6367 | 3 1 1 | 10 |
| 2 | 37.2783 | 2 2 2 | 7.46 |
| 3 | 62.9217 | 4 4 0 | 6.71 |
| 4 | 57.2938 | 5 1 1 | 6.68 |
| 5 | 74.4585 | 5 3 3 | 6.57 |
| 6 | 30.2538 | 2 2 0 | 9.29 |
| | | Average | 8 nm |

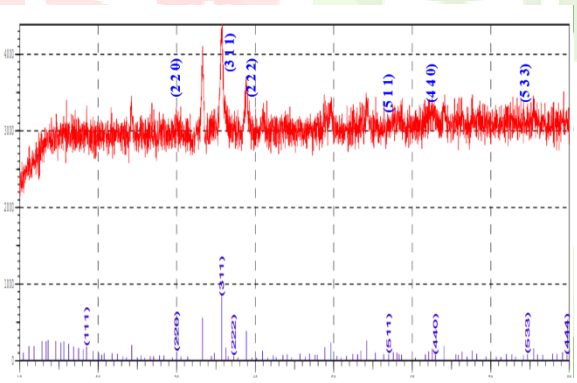

**Fig.3.** XRD peak profile for synthesized copper ferrites powder at 500°C

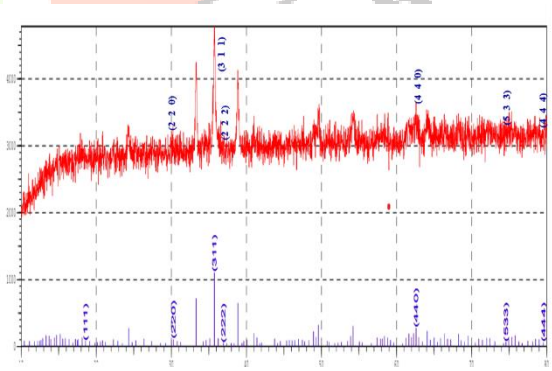

**Fig.4**. XRD peak profile for synthesized copper ferrites powder at 600°C

### 3.2 FTIR analysis

FTIR spectroscopy is applied to measure the chemical functional group analysis and absorption of mid-range IR energy started from 400 $cm^{-1}$ to 4000 $cm^{-1}$by studied samples. The FTIR analysis demonstrated the functional groups presented on synthesized copper ferrites at different calcination temperatures. The spectrum profile of prepared samples exhibited some characteristic bands related to physical and chemical variations. According to the FTIR results, 2162.86 $cm^{-1}$, 2051.21 $cm^{-1}$, 1980.95 $cm^{-1}$, 2324.88 $cm^{-1}$, 2051.38 $cm^{-1}$, and 1980.78 $cm^{-1}$ absorption peaks attributed to C-O stretching mode. The absorption bands between 522.12 $cm^{-1}$ and 420.22$cm^{-1}$ attributed to Fe–O and Cu–O ferrites and oxygen single bond stretching mode. All in all, most of the absorptions bands in respective samples are due to Fe–O and Cu–O stretching mode in ferrites characteristics. Table [C] showed the FTIR analysis data of synthesized copper ferrites at different calcination temperature.

Table 3.3 Observable wave number and functional group of synthesized copper ferrites calcined at 500°C and 600°C

| Temperature | Wave No ($cm^{-1}$) | Functional group |
|---|---|---|
| 500°C $CuFe_2O_4$ | 2162.86 | C-O stretching |
| | 2051.21 | C-O stretching |
| | 1980.95 | C-O stretching |
| | 522.12 | Fe-O and Cu-O |
| | 430.24 | Fe-O and Cu-O |
| | 420.22 | absorption bands of ferrite |
| 600°C $CuFe_2O_4$ | 2324.88 | C-O stretching |
| | 2051.38 | C-O stretching |
| | 1980.78 | C-O stretching |
| | 1560.65 | Non bonded O-H group |
| | 1114 | C-O stretching |
| | 513.89 | Fe-O and Cu-O |
| | 427.05 | Fe-O and Cu-O |
| | 419.58 | Cation oxygen Octahedral |

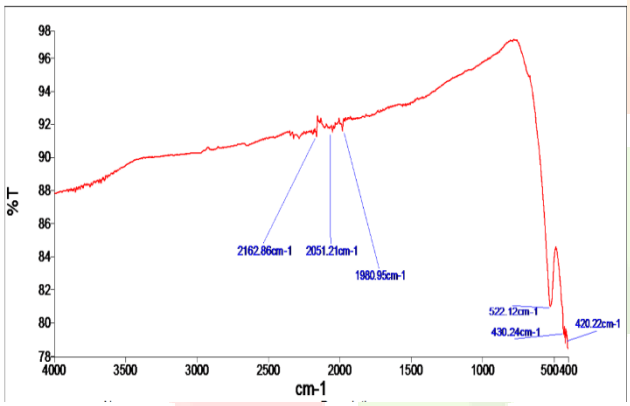


**Fig.5.** FTIR transmittance profile for synthesized copper ferrites powder at 500°C

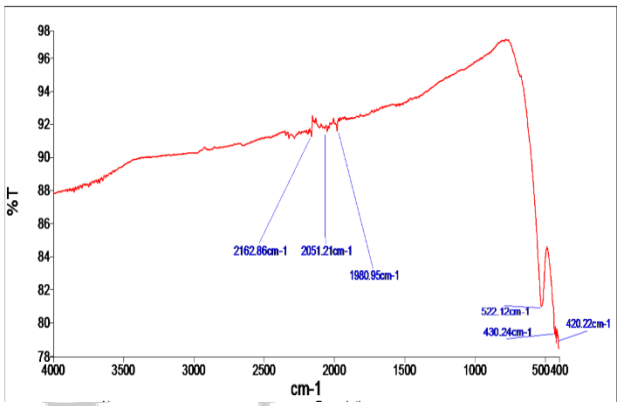


**Fig.6**. FTIR transmittance profile for synthesized copper ferrites powder at 600°C

### 3.3 Morphological analysis

Based on the SEM microstructural analysis, morphological nature of synthesized copper crystalline powder showed some are agglomerate, fine grain, spherical shape, crack free and dense surface. The average diameter and area distribution of synthesized copper ferrites with different calcination temperature are between (3.73 and 3.77) µm at 500°C and 600°C respectively. As a result, the morphologies and grain orientation of the synthesized copper ferrites crystalline powder are varied strongly and sharp with various calcination temperature.

Table 3.4 Comparison of the average grain size of synthesized copper ferrites at different calcination temperature

| SEM analysis data for $CuFe_2O_4$(500°C) | | | |
|---|---|---|---|
| **Min Grain** | **Max Grain** | **Average Grain size** | **Temperature (°C)** |
| 2.453µm | 5.424µm | 3.73µm | 500°C |
| 1.391µm | 5.807µm | 3.77µm | 600°C |

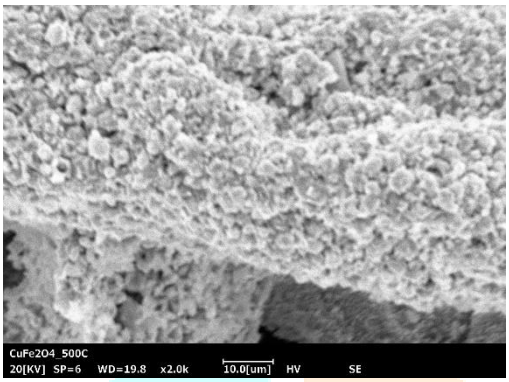


**Fig.7**. SEM image depicted for synthesized copper ferrites powder at 500°C

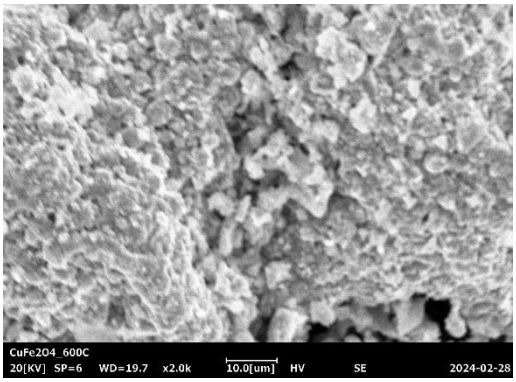


**Fig.8.** SEM image depicted for synthesized copper ferrites powder at 600°C

## IV. Conclusions

In this scenario, crystalline state synthesized $CuFe_2O_4$ nano-scale powder has been prepared by Sol-gel auto combustion technique. As-prepared $CuFe_2O_4$ nano-scale powder were calcined at different temperature at 500°C and 600°C separately. Interpretation from XRD peak profile showed that the average crystallite size of 500°C and 600°C were 7.7 nm and 8 nm respectively. According to the observable value of XRD data the crystallite size of prepared $CuFe_2O_4$ nano-scale powder were increased with increasing calcination temperature. Increasing the crystallite size of synthesized sample due to its merging process in solid state research field. Morphological nature of synthesized copper crystalline powder showed some are agglomerate, fine grain, spherical shape, crack free and dense surface. The average grain size with micron scale for synthesized $CuFe_2O_4$ were 3.73 and 3.77 µm. For the results of FTIR analysis, all of the absorption especially in region between 522.12 $cm^{-1}$ and 420.22 $cm^{-1}$were confirmed Fe–O and Cu–O ferrites function group formation. All in all, we may deduce that the behavior of functional group orientation, structural information and morphological nature of synthesized powder with different calcination temperature led to use in ferrites nanofibers, water treatment process and core application in inductor of electronic appliances.

## V. Acknowledgment

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