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ORIGINAL ARTICLE

# Synthesis and characterization of Ag doped Cobalt Ferrite nanocomposite

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**Abstract**
Nanomaterials are attracted a great deal of attention from scientific community due to its unique properties and applications. The small size ferrites have opened the door for intensive research to utilize their properties for biomedical applications. Cobalt ferrite nanomaterials and its silver doped (Ag-doped) nanocomposites have been prepared using solid state combustion method. This combustion method was carried out using polyvinyl alcohol (PVA) as a fuel for combustion reaction. The structure of the prepared cobalt ferrite and its silver nanocomposites were characterized by using X-ray diffraction (XRD) tool and morphology by Scanning Electron Micrograph (SEM) tool respectively. Bonding nature of the sample was studied by Fourier transfer infra-red (FT-IR) studies. Presence of the metals in the composites was confirmed by EDX pattern.

**Keywords:** *Ag Doped; Cobalt Ferrite; Energy Dispersive X-Rays (EDX); Fourier Transfer Infra-Red (FT-IR); Scanning Electron Micrograph (SEM); X-Ray Diffraction (XRD).*

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## INTRODUCTION

Nanomaterials are corner stones of nanoscience and nanotechnology which are broad and interdisciplinary area of research and developmental activity that has been growing explosively worldwide in the past few years. Nanotechnology is based on the science that deals with tweaking of the matter at the atomic and molecular scale and the size range between 1- 100 nm [1]. Recently researchers are attracted much towards nanomaterials due its enhanced properties and applications [2]. The latest physical properties and advancement in sample preparation of the materials at nanosize accounts the progress of nanoscience [3-4]. The man in his quest for knowledge has been conceiving and developing physical world and its components in bigger than the biggest and smaller than the smallest dimensions of mass, length and time [5]. Sometimes the changes in particles size are in such extent that the completely new transpiration is digging up which helps in flowering of the world [6-7]. However the title is know about how the biological activity takes place for certain materials when it is reduced into nanoscale dimensions. In this world of elaboration nanotechnology, one of the main primary concerns should be the potential environment impact of nanoparticles (Nps). A proper way of estimating the nanotoxicity is to monitor the response of the bacteria against these nanoparticles [8-10]. Applications being developed for nanomaterials include adding antibodies to nanotubes to form bacteria sensors, making a composite with nanotubes for aircraft, adding boron or gold to nanotubes to trap oil spills, include smaller transistors, coating nanotubes with silicon to make anodes the can increase the capacity of Li-ion batteries by up to 10 times. In addition to this, applications being developed for nanocomposites include a nanotube-polymer nanocomposite to form a scaffold which speeds up replacement of

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broken bones etc [11-15]. Transition metal ferrites, both doped and undoped, are magnetic candidates in a huge range of applications considering catalysis, sustainable hydrogen production application and electronic and magnetic devices, with others [11-15].

Cobalt ferrite material have attracted a great interest in fundamental and applied research due to their various properties like mechanical, hardness, thermal stability and anisotropy constant. All these characteristics encourage their use in wide range of applications from medicine to electronics. Relating to all these possible applications, different research groups performed various studies on the influence rare earth cations on the properties of $CoFe_2O_4$ in bulk form thin films and nanoparticles [16]. Cobalt ferrite as a magnetostrictive material has recently become of interest to many researchers due to its promising magnetostrictive properties. It has been proposed to be a suitable magnetostrictive material for some applications in the area of sensors and actuators. In such applications, speed of response with accurate displacements is important. Although cobalt ferrite has shown a small magnetostrictive coefficient, which is a disadvantage, it has also shown a very small hysteresis characteristic. It follows that, the results of using this material should lead to a smaller amount of energy loss and higher displacement accuracy at high frequencies, and these benefits may compensate this disadvantage [17].

The present study reports the synthesis of Ag metal, $CoFe_2O_3$ and Ag doped cobalt ferrite using combustion method through metal carboxylate as precursor. PVA is used as a fuel for the combustion process. Prepared samples are well characterised by various characterisation techniques such as XRD, SEM, IR, EDX etc.

## EXPERIMENTAL

*Materials and methods*

The chemicals like $AgNO_3$, oxalic acid, iron oxide, polyvinyl alcohol, Cobalt chloride chemicals are used are of AR grade. Combustion method is used for synthesis of sliver metal, ferrite materials and Ag-doped cobalt ferrite nanocomposites materials. Polyvinyl alcohol is used as fuel for combustion reaction.

*Preparation of metal oxalate*

The cobalt oxalate is prepared by dissolving known quantity of cobalt chloride and oxalic acid in 100 ml of double distilled water in a separate container with the molar ration of 5 : 5 respectively. These solutions are mixed together in a separate container and kept it on the magnetic stirrer for 1 hour to complete conversion of cobalt chloride into its oxalate by reacting with oxalic acid. Finally, as formed cobalt oxalate precipitation is washed with distilled water and dried by passing hot air with minimum pressure. Similar procedure is adopted for the preparation of iron oxalate and silver oxalate using respective metal salts such as ferrous ammonium sulphate and silver nitrate respectively by reacting with oxalic acid.

*Synthesis of nanosized silver (Ag) metal*

In the typical combustion process, the silver oxalate is burned with the poly (vinyl alcohol) (PVA) as a fuel with the molar ratio of 1: 5; the combustion reaction is arrested at the particular temperature for the phase formation. The impurities present in the final product are separated by treating with acetone and dried.

*Synthesis of $CoFe_2O_4$ nanoparticles*

Cobalt ferrites nanomaterials are prepared by burning cobalt oxalate with iron oxalate using poly (vinyl alcohol) as a fuel for the combustion reaction in 1: 5 ratio. The burning process takes

$CoO + Fe_2O_3$------------------ $CoFe_2O_4$

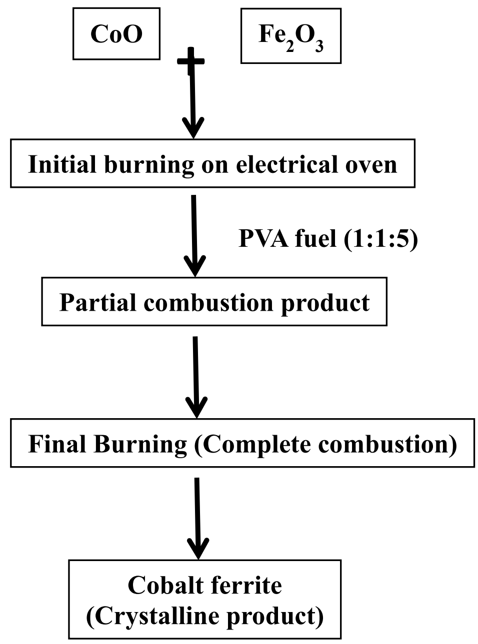


Fig. 1. Schematic flow of cobalt ferrite synthesis.

place in a separate container to the particular temperature which results into the possible formation of partial metal oxides *i.e.*( CoO and $Fe_2O_3$). These oxides are mixed properly with PVA, grinded and burned again in a separate container at ratio 1: 1: 5, respectively. This process starts with formation of froth, evolution fumes and burns with flame; finally it was completed at particular temperature. The product cobalt ferrite obtained after the complete combustion process and is grinded in the pestle and motor for half an hour. It is washed with acetone and concentrated action to get rid of carbon particles in the final product. Following is the possible reaction taking place during combustion and synthetic method is given in below Fig. 1.

*Synthesis of Ag doped ferrite nanomaterials*

Cobalt ferrite nanomaterials are doped with the silver metal nanoparticles using the combustion method. The process starts with burning of cobalt ferrite with silver metal nanoparticles, and PVA keeping the molar ratio as 2: 1: 5, respectively. Initially, it burns with sooty flame followed by reduced non sooty flame for complete reaction. This reaction was arrested at a particular temperature around 500⁰C to form the phase formed Ag doped cobalt ferrite nanoparticles. Later it is washed with acetone and concentrated action to remove the carbon particles and other organic impurity.

## RESULTS AND DISCUSSION

*X-ray Diffraction (XRD)*

The crystalline nature of the synthesized nanoparticles was analyzed by X-ray diffraction tool. Fig. 2 shows XRD pattern of the synthesized nanoparticles after the reduction of silver nitrate into the silver metal nanoparticles. Intense peaks corresponding to (111), (200), (220) and (311) were observed for the silver nanoparticles in accordance with JCPDS files no. 03-0921. This

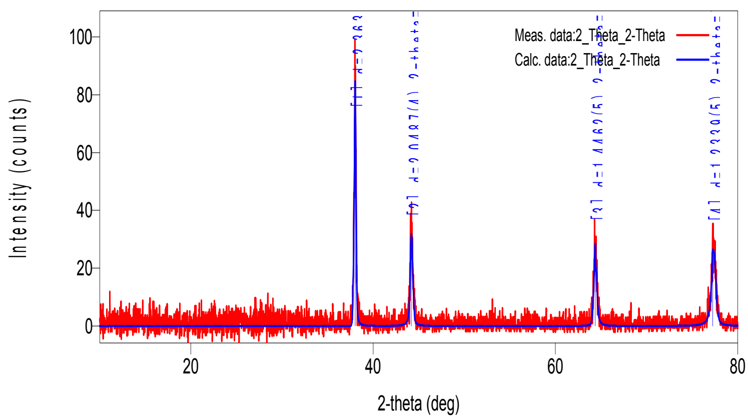


Fig. 2. XRD pattern for Silver nanoparticles.

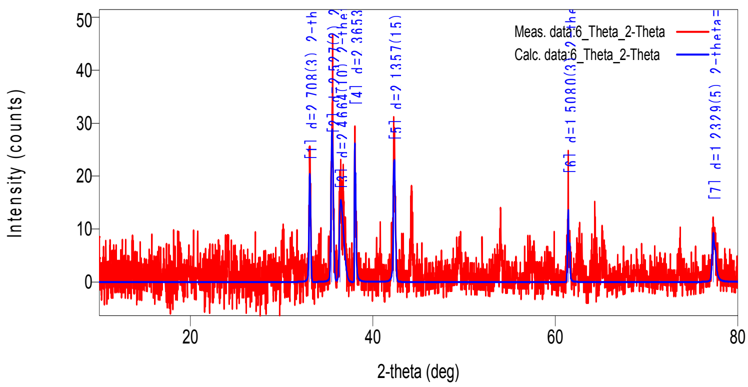


Fig. 3. XRD patterns for Ag doped $CoFe_2O_4$.

confirms the formation of crystalline silver metal nanoparticles.

The Fig. 3 shows the XRD pattern of the as prepared silver doped $CoFe_2O_4$, nanomaterials. This pattern shows the Bragg's reflection which indicates the presence of the crystalline nature of the above samples. Peaks obtained in the pattern are matched with standard JCPDS file of the silver metal and $CoFe_2O_4$ (22-1086) nanomaterials. The above pattern represents the presence of the silver doped in $CoFe_2O_4$. These results are supported by the EDX results.

*Scanning Electron Microscope (SEM)*

Field Emission Scanning Electron Microscope has been used to study the particle morphology. Fig. 4 a shows SEM images of prepared Ag metal at low and high resolution. This image shows silver metal nanoparticles, size ranging in nanometer and has the irregular shaped particles forms a compact sheet like arrangement can be observed [18]. The clear image can be observed in higher resolution image as in Fig. 4 b.

Fig. 4 c shows the SEM images of prepared silver doped $CoFe_2O_4$ nanomaterials at low and high resolution respectively. Microstructure image shows particles are in self dispersed micro image structure. Some particles are higher densed and form very compact morphology. The clear compact image is viewed in the higher resolution as in Fig. 4 d.

The image shows almost particles are spherical in nature and same particles shows micro-self-doping forms a micro injecting. This kind of morphology will be highly applicable in the catalysis. Micro-self doped can be viewed properly in the high resolution image.

*Energy dispersive X-Rays (EDX)*

EDX (energy-dispersive X-ray) report confirms a chemical composition or contaminants for the synthesized silver doped ferrites nanomaterials. Fig. 5 shows EDX pattern of as prepared Ag metal. The pattern shows Ag highest percentage signals at respective position confirms the formation of Ag metal.

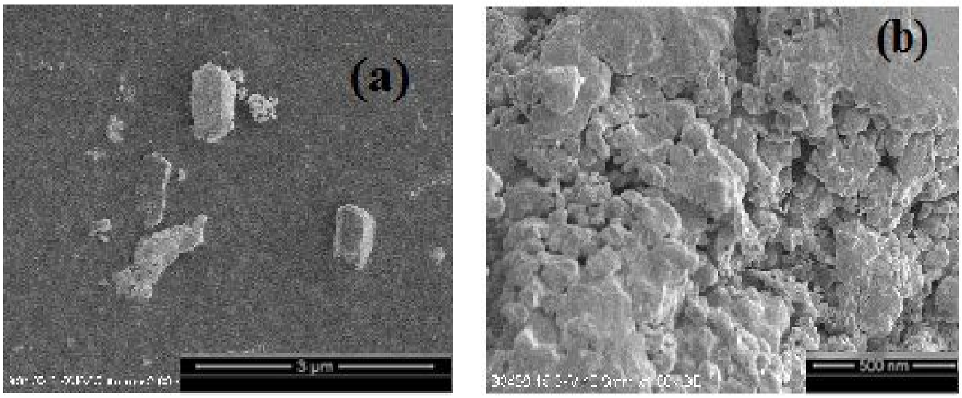


Fig. 4 a, 4 b .SEM images of as prepared silver nanoparticles with high and low resolution.

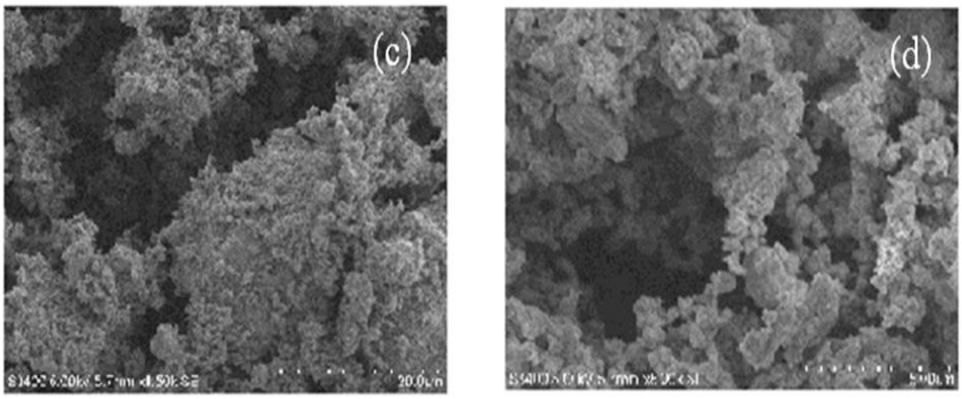


Fig. 4 c, 4d. SEM image of as prepared silver doped $CoFe_2O_4$ nanomaterials with high and low resolution.

Fig. 6 shows the EDX pattern of Ag doped cobalt ferrite. The EDX spectrum gives the highest percentage signals of cobalt, silver and iron for contaminants in nanoparticles. Results of our study prove that there are some contaminants which are showed in the Table 1. This confirms the doping of Ag metal with cobalt ferrite.

*Infrared Study (FT-IR)*

Table 2 shows vibrational frequencies of as prepared Ag doped cobalt ferrite. The sample shows peaks at 3250, 1580, 1050, 680, 545, 400 $cm^{-1}$. The metal-oxygen bonding and nature of the synthesized oxide sample was carried out by infrared study. Metal oxides generally give absorption bands below 1000 $cm^{-1}$ arising from inter-atomic vibrations [19]. The peak 3250 $cm^{-1}$ corresponds to water of absorption. Vibrational frequency at 1580 $cm^{-1}$ is due to the presence of carbon dioxide. Frequency at 1050 $cm^{-1}$ is due to some overtones. Peaks below 1000 $cm^{-1}$

Table 1. Metal indexing of the Ag metal and Cobalt ferrite by EDX pattern.

| Samples | Metal indexing (keV) |
|---|---|
| Ag | Ag(2.8), Ag(3.0) |
| Ag-$CoFe_2O_4$ | Ag(0.2), Co(0.4), Fe(0.4), Co(0.7), Fe(6.4), Co(6.9), Fe(6.9), Co(7.7) |

Table 2. Vibrational frequencies of Ag-doped Cobalt ferrite.

| Sl. No | Frequencies($cm^{-1}$) |
|---|---|
| 1 | 3250 |
| 2 | 1580 |
| 3 | 1050 |
| 4 | 680 |
| 5 | 545 |
| 6 | 400 |

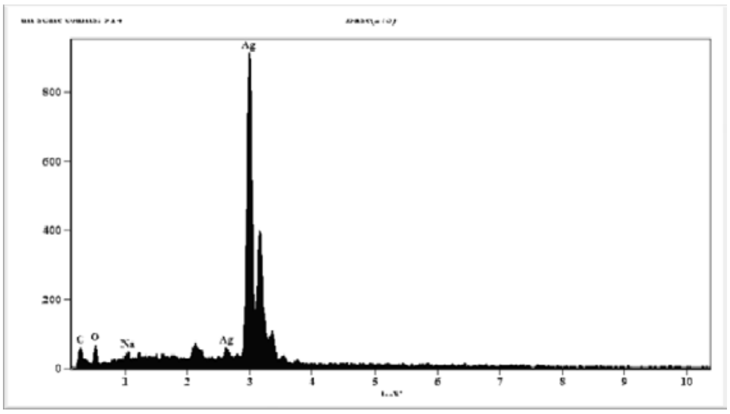


Fig. 5. EDX images of silver metal nanoparticles.

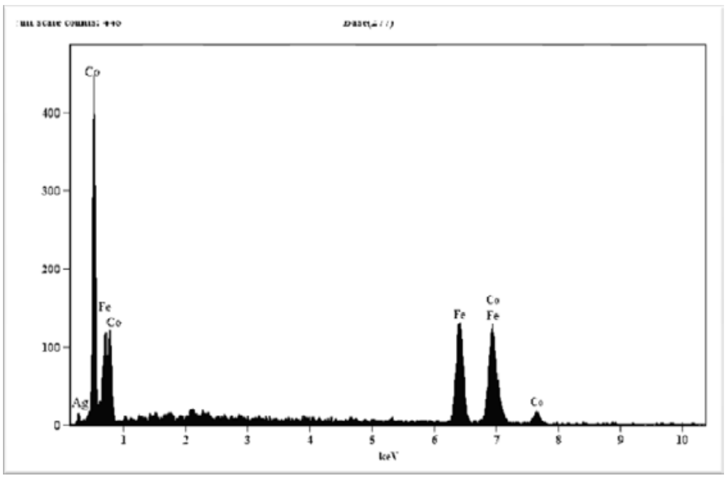


Fig. 6. EDX images of Ag doped $CoFe_2O_4$.

corresponds to Metal-oxygen (M-O) vibrational modes of the samples conform the formation of Ag doping cobalt ferrite [20].

## CONCLUSIONS

Following are the conclusions made by our study

1) Combustion method for the reduction of Ag metal from silver salt, cobalt ferrite and Ag doped cobalt ferrite is very simple and it needs simple equipments.
2) This method may be used for the reduction of other metals from its compounds and metal oxides from metal salts or metals.
3) PVA is acting as a good fuel for the combustion process. Initially it froths and forms the expected solid product after complete combustion.
4) This method insists for the doping of other metal to the any ferrites and other nanomaterials.

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## CONFLICT OF INTEREST

The authors declare that there are no conflicts of interest regarding the publication of this manuscript.

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