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Design and investigation of Sono-chemical synthesis of pure and Sn doped CoFe₂O₄ nanoparticles and their structural and magnetic properties



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ABSTRACT

Research on the tuning of magnetic properties of the divalent ferrite materials is always having its own flavor due to their spectacular applications in the modern electronic and magnetic devices including magnetic data storage and magnetic tapes. In this present context, we have reported the sono-chemical synthesis of pure and Sn doped $CoFe_2O_4$ nanoparticles (NPs) and their structural, morphological and magnetic properties. Powder X-ray diffractometry (XRD) and scanning electronic microscopic (SEM) techniques have provide the clear details about the formation crystallographic structure and surface morphological features and obtained the XRD results showed that the stable cubic crystallographic structure and stable hard ferromagnetic state even at ultrasonic waves irradiated conditions. On the other hand, Sn doped $CoFe_2O_4$ has high coercivity (1621 Oe) compared than the pure $CoFe_2O_4$ and few technologically important doped $CoFe_2O_4$ important ions such as Gd^{3+} , Tm^{3+} , Eu^{2+} in $CoFe_2O_4$ NPs respectively and hence the Sn doped $CoFe_2O_4$ NPs is suggested the magnetic data storage applications.

1. Introduction

Functional ferrite materials have widely used in the technologically important applications particularly in microwave devices, magnetic recording, magnetic imaging and magnetic data storage due to its high saturation magnetization with low eddy current losses. Divalent ferrite materials (MFe₂O₄, M: Zn, Co, Ni, Cu, Mn, Mg) are classified in two categories such as inverse and normal spinel and the spinel type is played a non-debatable role in the ferrite materials physical and chemical properties [1-4]. In addition to that, both spinel type ferrites materials have falls under the category of semiconductor ceramics which are possesses high electro-magnetic performance including high dielectric constant and high saturation magnetization [5–6]. Among the above-mentioned divalent ferrite materials, CoFe₂O₄ is one of the leading ferrite material for the aforementioned applications and so far numerous nano-science researchers have been found verity of physicchemical and magnetic properties while deal with the synthesis process, particle size, shape and spinel type structural aspects [7-10]. In addition to that, incorporation of the divalent and trivalent metal ions in to the spinel ferrites systems also has a considerable interest due its capable of the modifying the functional properties particularly electrical and magnetic properties [11–15]. For better understanding, here we have listed out few divalent and trivalent metal ions doped CoFe₂O₄ spinel ferrites and their functional properties. Atta ur Rahman et al., have been investigated the dielectric constant and electrical conductivity for the pure and Sn-doped nanocrystalline CoFe₂O₄ and found Sn doped CoFe₂O₄ has high electrical conductivity and dielectric constant [16]. Raghvendra Singh Yadav et al., have been reported the sonochemical synthesis of pure and Gd^{3+} doped $CoFe_2O_4$ and found the coercivity values are 234.32 Oe and 12.60 Oe respectively [17]. Similarly, the pure and Tm³⁺ substituted cobalt ferrite is synthesized by sonochemical approach and found the coercivity values are 15.06 Oe and 20.23 Oe respectively [18]. Ngema et al., have been demonstrated the synthesis and magnetic properties of pure and Sn-Doped CoFe₂O₄ and found considerable enrichment of the coercivity value and the observed values are 73.2 and 89.1 Oe respectively at room temperature

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Received 18 January 2023; Received in revised form 19 June 2023; Accepted 2 July 2023 Available online 3 July 2023 1387-7003/© 2023 Published by Elsevier B.V. [19]. Based on the overall literature reports on the divalent and trivalent ions doped $CoFe_2O_4$ NPs, we have found that, Sn doped $CoFe_2O_4$ structural, magnetic and electrical properties are still not clearly understood and the best of our knowledge, we have not found any reports of sono-chemical synthesis of the Sn doped $CoFe_2O_4$ NPs. In addition to that, based on the above-mentioned reports, it is clear that, the incorporation metal ions in the ferrite's structures have played a vital role in the structural and magnetic behaviors and we need to have an essential knowledge about the above-mentioned properties for developing the advanced devices for high frequency microwave, electronic devices and magnetic applications.

In the present work, we have reported the pure and Sn doped $CoFe_2O_4$ NPs with a sonochemical-assisted approach. Sn doped $CoFe_2O_4$ NPs have high coercivity compared to the pure $CoFe_2O_4$ and no significant changes have been found for the ultrasonic waves irradiated samples.

2. Experimental procedure

Co ferrites were synthesized from Cobalt Nitrate Nona hydrate (Co (NO₃)₂·6H₂O), Sigma-Aldrich purity: 99.95%) and Ferric nitrate Nona hydrate (Fe (NO₃)₃·9H₂O) Sigma-Aldrich purity: 99.95%), Stannous Chloride Trihydrate (SnCl₂·3H₂O) utilizing co-precipitation method and these kind of nitrate materials are highly suitable to synthesis the pure cubic ferrite materials [4,20]. The proper stoichiometry proportions (0.5 M of Cobalt Nitrate and 1 M of Iron Nitrate) of metal nitrate salts are dissolved in D.I water. The proper amount of urea (CO(NH₂)₂) (Sigma-Aldrich purity: 99.90%) is added to the above solution which is act as surfactant agent. The solution is continuously stirred on 70 degreesC hot plate for 2 h. The precipitation has carried out using by adding of NaOH in to the solution. The precipitate was filtered, dried and sintered at 800°C for two hours. For the synthesis of Sn_{0.2}Co_{0.8}Fe₂O₄ sample, the proper stoichiometry amounts of stannous chloride were added to the cobalt and ferric nitrate mixed aqueous solution then followed the same way as described above for the cobalt non - substituted ferrite. Furthermore, similar process has been followed for the sonochemical approach synthesis [ref] of pure and Sn doped CoFe₂O₄ NPs. The prepared mixed solutions (pure and doped CoFe₂O₄) were to high-intensity ultrasonic irradiation (Ultrasonic homogenizer UZ SONOPULS HD 2070; frequency: 20 kHz and power: 70 W) for 60 min. At the end of the ultrasonication, the reaction temperature was measured as 90 degreesC owing to high number of collisions between the reactants. The received product was washed several times with DI water. The magnetic solid product was separated from the liquid by simple external magnet. It was dried at 60 degreesC for 12 h. The prepared nanoparticles were separated by centrifuge process. The obtained nanoparticles were washed several times in deionized water for complete removal of sodium ions. Then, the obtained washed nanoparticles were dried at 60 degreesC for 24 h.

3. Results and discussion

3.1. Structural properties

Powder X-ray diffractometry technique has been utilized to examine the structural features of the pure, Sn doped and ultrasonicated $CoFe_2O_4$ NPs and the recorded XRD profiles have showed in Fig. 1. As seen in Fig. 1a, the pure $CoFe_2O_4$ NPs XRD pattern is well matched with the previous literature [20] as well as CCDC data base (CIF: 109044). Based on the observed diffraction lines positions, it is clear that the pure $CoFe_2O_4$ NPs are crystallized in the cubic spinel ferrite crystallographic structure and the indexed crystalline planes are as follows: (220), (311), (222), (400), (422), (511), (440) and (533) respectively [20]. Note that, in the case of the pure $CoFe_2O_4$ NPs, there is no mixed iron oxide phase observed. The broad crystalline peak features illustrate that the synthesized pure $CoFe_2O_4$ NPs belong to the nano-crystalline regime.



Fig. 1. (a) XRD patterns of the pure, Sn doped and ultrasonicated CoFe₂O₄ NPs.

In the case of ultrasonic waves irradiated conditions, all the $CoFe_2O_4$ NPs XRD crystalline peaks are re-appeared and no peak distortion and deformations have been found and hence it could be confirmed that the cubic spinel ferrite phase is crystallographically stable at ultrasonic waves irradiated conditions [20].

Note that, as per the Ref [20], similar kind of CoFe₂O₄ NPs structural response has been examined at supersonic shock wave irradiated conditions and found the occurrence of the significant structural deformations. At supersonic shocked conditions, the major diffraction plane (311) is experienced the lower angle shift from 35.53 to 35.13 due to the occurrence of the significant structural deformations such as degree of crystalline nature, elastic/plastic deformations, crystallite size, lattice unit expansion, cation disorder and bond length [20]. But in the present case, we could not able to see such structural deformations at ultrasonic waves irradiated conditions. For example, the peak major diffraction crystalline peaks such as (220) and (311) diffraction peak positions and full-width maximum values have been presented in Table.1. Based on the diffraction peak position and intensity profiles, there are no evidences found for the ion migration from A site to B site during the ultrasonic waves irradiated conditions. On the other hand, in the case of Sn doped CoFe₂O₄ NPs, all the diffraction lines are reproduced as like that of the pure CoFe₂O₄ NPs and we could be able to see the appearance of the diffraction line at 26.5 degree (110) and 51.8 degree (211) respectively and both planes are belonging to SnO₂ crystal structure for better understanding we have presented the zoomed-in XRD patterns from 25 to 37 degree in Fig. 2. The prominent diffraction peak (311) is located at 35.520 degrees for the pure CoFe₂O₄ NPs and this diffraction angle position remains unchanged for the Ultrasonicated CoFe₂O₄. Sn doped CoFe₂O₄ and Ultrasonicated Sn doped CoFe₂O₄ and the required vales are presented in Table.1. Followed by the diffraction peak position, the diffraction peak intensity is another important parameter to describe the degree of crystalline nature of the samples during the irradiation and chemical doping conditions. The calculated normalized intensity of the (311) plane of the pure CoFe₂O₄. Ultrasonicated CoFe₂O₄. Sn doped CoFe₂O₄ and Ultrasonicated Sn doped CoFe₂O₄ values are as follows: 0.915, 0.915, 0.888, and 0.922 respectively. The calculated average grain size values for the pure CoFe₂O₄. Ultrasonicated CoFe₂O₄. Sn doped CoFe₂O₄ and Ultrasonicated Sn doped CoFe₂O₄ are 26, 28, 27 and 27 nm respectively.

In the case of Sn chemical doping, there is slight diffraction angle shift observed at (220) and (311) planes which clearly represent the occurrence of the Sn ions in the cubic spinel ferrite B-site. Moreover, we can be able to see the formation of two diffraction lines at 33.0 and 33.9

Table 1

Structural feature of the pure, Sn doped and ultrasonicated CoFe₂O₄ NPs.

Name of the synthesized NPs CoFe ₂ O ₄	(311)			(220)		
	Diffraction position (deg)	Peak height	FWHM	Diffraction position	Peak height	FWHM
Pure CoFe ₂ O ₄	35.520	0.915	0.353	30.156	0.2384	0.3205
Ultrasonicated CoFe ₂ O ₄	35.532	0.915	0.294	30.170	0.2106	0.2371
Sn doped CoFe ₂ O ₄	35.489	0.888	0.319	30.134	0.1868	0.2830
Ultrasonicated Sn doped CoFe2O4	35.483	0.922	0.312	30.122	0.2087	0.2995



Fig. 2. Zoomed in XRD patterns of the XRD patterns of the pure, Sn doped and ultrasonicated CoFe₂O₄ NPs.

degree and these diffraction lines belongs to iron oxide phase (α -Fe₂O₃) the corresponding crystalline planes are (104) and (110) respectively [18]. The formation of the α -Fe₂O₃ phase in the ferrite materials as a secondary phase is quite usual and during the doped conditions most of researchers have been found such kind of the α -Fe₂O₃ phase as secondary phase [18]. Due to the formation of the secondary phase and incorporation of doped ions in the cubic crystal system, considerable changes have been expected in the magnetic and electrical properties of the ferrite material. In the case of ultrasonicated irradiation conditions, the Sn doped CoFe₂O₄ NPs do not showed any crystallographic phase transitions and ion migration from A-site to B-site and vice versa. As like that of pure CoFe₂O₄ NPs, the Sn doped CoFe₂O₄ NPs are also having a stable crystal system at ultrasonic waves irradiated conditions.

4. Surface morphological studies

In the case of the nano regime, understanding of the particle size and shapes are very important to better comprehending the functional properties such as magnetic and electrical properties [21–22]. Hence, we have performed morphological aspect of investigations by scanning electronic microscopic (SEM) technique and the captured micrographs are presented in Fig. 3.

The pure $CoFe_2O_4$ NPs (Fig. 3a) showed the sphere shapes and size of the $CoFe_2O_4$ NPs particles is varied from 287 to 57 nm wherein the estimated average particle size is 159 nm. In the case of ultra-sonic wave irradiation conditions, there is no remarkable changes have been

noticed and the calculated particles size is varied from 351 to 98 nm and the obtained average particle size is 157 nm. Based on the morphological aspects of investigations, the test material not undergone any surface related modifications at ultra-sonic wave irradiation conditions and it is well matched with X-ray diffraction results as well. In the case of Sn doped CoFe₂O₄ NPs SEM images (Fig. 3c), we could be able to see the well definite shape of the particles and agglomeration is also very less while compared to the pure CoFe₂O₄ NPs and found the average particle is 138 nm and the ultrasonicated Sn doped CoFe₂O₄ NPs particle size is 120 nm. In the case of doped ferrite samples, particle size is considerably reduced by the impact of the ultrasonic waves.

5. Magnetic properties

Even though magnetism in solid state materials has enough understanding in several decays, in the case of ferrite nano materials understanding of the magnetic properties are still has its own essence due to the showing of outstanding magnetic properties within the cubic systems. Moreover the spintronics device engineers are always searching outstanding magnetic ferrite materials for the device making applications and due to this strong technological aspect of requirement, investigations on magnetic properties on the ferrite materials are greatly required. Hence, we have performed the magnetic properties of the above mentioned NPs at room temperature by vibrating sample magnetometer (VSM) and obtained VSM loops are presented in Fig. 4. As seen in Fig. 4, the pure CoFe₂O₄ NPs showed the hard ferromagnetic



Fig. 3. SEM images of the CoFe₂O₄ NPs (a) pure CoFe₂O₄ NPs (b) Ultrasonicated CoFe₂O₄ NPs (c) Sn doped CoFe₂O₄ NPs (d) Ultrasonicted Sn doped CoFe₂O₄ NPs.



Fig. 4. M-H loops of the pure, Sn doped and ultrasonicated CoFe₂O₄ NPs.

behavior with the Coercivity value is 1036 Oe and the saturation magnetization is 71 emu/g [20,22]. Based on the diffraction and microscopic results, we would be expected the stable magnetic state at ultrasonic waves irradiated conditions. As we expected, at ultrasonic waves irradiated conditions, the hard ferromagnetic state exists with the Coercivity value 963 Oe and the saturation magnetization value is 68 emu/g. Based on the observed Coercivity and saturation magnetization results, the ultrasonic waves irradiated CoFe₂O₄ NPs numerical values are quite low, but the observed changes are not significant [20]. Hence, we can consider as a stable magnetic state at ultrasonic waves irradiated conditions. Note that, in the case of super-sonic shock waves loaded conditions, the CoFe₂O₄ NPs hard ferromagantic state is converted in to the super-paramagnetic state due to the occurrence of lattice distortions [20]. Note that, the saturation magnetization values of the control and supersonic waves irradiated conditions are 76.5 and 40 emu/g respectively [20].

In addition to that, the Coercivity values of the control and supersonic shock waves irradiated samples are 910 and 14 respectively and this massive reduction of the Coercivity due to the shock wave induced significant modification of the crystal lattice, particle size reduction and enhancement of hopping length between A site and B-site [20]. But, at ultrasonic waves irradiated conditions, there is no such kind of remarkable reduction of saturation magnetization and Coercivity has been noticed and which clearly demonstrated the stable magnetic state (Fig. 4) and crystallographic structure (Fig. 1). In the case of Sn doped conditions, Sn doped CoFe₂O₄ NPs are also showed the ferromagnetic state and with the high Coercivity value is 1621 Oe and the saturation magnetization is 59 emu/g and ultrasonic waves irradiated Sn doped $CoFe_2O_4$ NPs have the Coercivity value is 1503 Oe and the saturation magnetization is 56 emu/g. As per the M-H loop area, Coercivity and saturation magnetization values, it is clear that, the Sn doped CoFe₂O₄ NPs magnetic state is also stable as like that of the pure CoFe₂O₄ NPs and it is clearly demonstrated that the stable magnetic state at ultrasonic irradiated conditions. Coercivity and saturation magnetization values of the pure, Sn doped and ultrasonic waves irradiated samples are presented in Table.2. On the other hand, while compared to the pure CoFe₂O₄ NPs, Sn doped CoFe₂O₄ NPs Coercivity value is significantly higher and the values are 1036 and 1621 Oe respectively and the possible reasons for the rapid enhancement of the Coercivity values are as follows. Based on the conventional bivalent and trivalent ions occupancy in the A (tetrahedral sites) and B site (octahedral sites) as follows. In the cubic spinel ferrite, Tin cations (Sn^{4+} and Sn^{2+}) have the higher tendency to occupy the B sites as while compared to the A sites [19,23-26]. The Sn⁴⁺and Sn²⁺ cations occupying octahedral sites govern the most transport phenomena properties such as polarization and conductivity of the materials due to their close proximity. The major advantage of the Sn doped CoFe₂O₄ NPs, Sn ion can occur in the B-sites two states such as Sn^{4+} and Sn^{2+} and they can be easily replaced the Co^{2+} ions in the same B-site and due to the existence of double valance state of tin ions (Sn⁴⁺and Sn²⁺) causes the fast electron exchange between these cations and found remarkable changes in the magnetic and electronic properties [19,25-26]. For example, Atta ur Rahman et al., have been demonstrated the Sn doped CoFe₂O₄ NPs showed the higher dielectric constant compared than the pure CoFe₂O₄ NPs [16]. Ngema et al., have systematically showed the changes in the saturation magnetization and coercivity values with respect to Sn ion concentration from 0.1 to 0.5 and found the increment of the coercivity and decrement of the saturation magnetization [19].

On the other hand, the divalent and trivalent ions have significantly reduced the coercivity of the $CoFe_2O_4$ NPs and here we have listed out few examples for the better understanding the changes and the values are presented in Table.3. The incorporation of the Gd^{3+} in $CoFe_2O_4$ system experiences the significant loss of coercivity values while compared to the pure $CoFe_2O_4$ and the values are 234.32 Oe and 12.60 Oe for the pure and Gd^{3+} in $CoFe_2O_4$ NPs [17]. The higher coercivity values are highly required for the magnetic data storage and spintronics

Table 2

Saturation magnetization and Coercivity of the pure, Sn doped and ultrasonicated $CoFe_2O_4$ NPs.

Name of the synthesized NPs CoFe ₂ O ₄	Saturation Magnetization (emu/g)	Coercivity (Oe)
Pure CoFe ₂ O ₄	71	1036
Ultrasonicated CoFe ₂ O ₄	68	963
Sn doped CoFe ₂ O ₄	59	1621
Ultrasonicated Sn doped CoFe ₂ O ₄	56	1503

Table 3

Comparative data for the Coercivity of the Sn doped and ultrasonicated $CoFe_2O_4$ NPs with few reported doped $CoFe_2O_4$ NPs.

Name of the synthesized NPs $XCoFe_2O_4$	Coercivity (Hc) at room temperature	Reference
Gd doped CoFe ₂ O ₄	12.60 Oe	[17]
Tm doped CoFe ₂ O ₄	20.23 Oe	[18]
Sn doped CoFe ₂ O ₄ (x-0.1)	89.1 Oe	[19]
Sn doped CoFe ₂ O ₄ (x-0.5)	270 Oe	[19]
Eu doped CoFe ₂ O ₄	937 Oe	[18]
Ultrasonicated Sn doped CoFe ₂ O ₄ (x-0.2)	1503 Oe	Present work
Sn doped CoFe ₂ O ₄ (x-0.2)	1621 Oe	Present work

applications. In that aspect, pure and Sn doped $CoFe_2O_4$ NPs are the potential candidates for the above motioned applications and moreover the Sn doped $CoFe_2O_4$ NPs showed the outstanding high coercivity value compared than the few technologically important ions doped $CoFe_2O_4$ and they are Gd^{3+} in $CoFe_2O_4$ NPs, Tm^{3+} in $CoFe_2O_4$ NPs, Eu^{2+} in $CoFe_2O_4$ NPs [17,18,].

6. Conclusion

As a summary of the present investigation, we have studied the impact of the ultrasonic waves on the pure and Sn doped CoFe₂O₄ NPs and their structural, morphological and magnetic properties have been examined and presented. The obtained XRD patterns of the ultrasonic waves irradiated pure and Sn doped CoFe₂O₄ NPs showed the stable crystallographic phase and there is no evidence for the structural deformations and structural transitions. SEM images are also showed the stable morphology as well as size of the particles at irradiated conditions and resultant the test samples do not undergo any surface and size related property changes. As a cumulative evidences of the abovementioned claims, the pure and Sn doped CoFe2O4 NPs saturation magnetization and Coercivity values are not significantly changed at ultrasonic waves irradiated conditions. The stable magnetic property illustrated that the applied ultrasonic waves do not induce any ion migration in-between A -site and B-site in the cubic spinel ferrite. Based on the overall figure out of the test samples results, the pure and Sn doped CoFe₂O₄ NPs are strongly suggested to the magnetic data storage applications due to its high saturation magnetization and Coercivity values.

Declaration of Competing Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

Data availability

No data was used for the research described in the article.

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