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Induction Heating Analysis of Surface-Functionalized Nanoscale CoFe_2O_4 for Magnetic Fluid Hyperthermia toward Noninvasive Cancer Treatment

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✔ **Cite this:** *ACS Omega* 2020, 5, 36, 23378–23384

Publication Date: September 2, 2020

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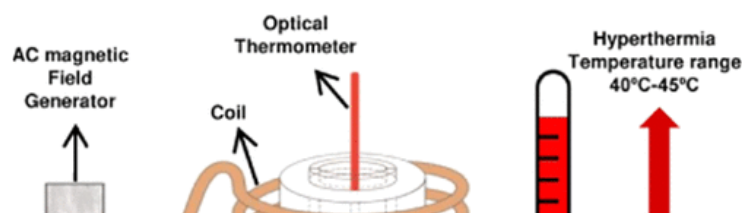
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Abstract



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route and characterized by standard techniques for structure, morphology, and magnetic properties analysis. The Rietveld refined X-ray diffraction (XRD) pattern of CoFe_2O_4 nanoparticles indicated the formation of a cubic-spinel single-phase structure with the $Fd\bar{3}m$ space group. The average crystallite size (~ 12 nm) confirmed the nanocrystalline appearance of the prepared CoFe_2O_4 nanoparticles. Transmission electron microscopy (TEM) images revealed the spherical nature of both (CoFe_2O_4) and (OA- CoFe_2O_4) samples. The absorption bands in the Fourier transform infrared (FT-IR) spectrum at $\sim 3418, 3026, 1628, 1404, 1068, 845, 544,$ and 363 cm^{-1} affirmed the spinel ferrite formation and OA attachment. The M–H curve recorded at room temperature showed the superparamagnetic nature of the CoFe_2O_4 nanoparticles with moderate saturation magnetization (~ 78 emu/gm). The blocking temperature of the prepared CoFe_2O_4 nanoparticles obtained from the field-cooled and zero-field-cooled (FC–ZFC) curve was estimated to be 144 K. Further, the characterized surface-modified CoFe_2O_4 was then added in ethylene glycol/water with various concentrations and characterized by the induction heating technique for the evaluation of their self-heating characteristics. A series of temperature versus time measurements were made by varying the ethylene glycol/water proportion for better understanding of the self-heating characteristics of the prepared CoFe_2O_4 nanoparticles. All of the findings display the applicability of the surface-modified CoFe_2O_4 nanoparticles in magnetic fluid hyperthermia toward noninvasive cancer treatment and other bio-applications.

1. Introduction

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Magnetic nanomaterials with appropriate physicochemical properties, functionalized surface, and improved biocompatibility have been broadly examined for different restorative, indicative, and remedial applications, for example, specific drug delivery, fluid hyperthermia, as a contrast enhancer in magnetic resonance or reverberation imaging (MRI), cell detachment, tissue fixing, biosensors, and so forth. (1–6) The physicochemical properties, colloidal strength, and biocompatibility of these magnetic nanomaterials can be vitally constrained by precise synthesis conditions and functionalization of the surface. Magnetic platforms with nanometric size (of the order of a few nanometers) and thin size distribution with upgraded magnetic properties comprise the prime necessity for biomedical application purposes. Likewise, magnetic nanoparticles should also have consolidated high susceptibility for the best magnetic enhancement and quicker loss of magnetization after disposal of the outer guest magnetic field. Magnetic fluid hyperthermia, a modular strategy for disease treatment with the desired temperature, in the range of $41\text{--}45\text{ }^\circ\text{C}$, and a treatment period of least 20 min, has been paid great attention to because of its lower clinical symptoms and probability to obliterate particular territory of harmful malignancy tumor. (7–9) This procedure includes the consolidation of superparamagnetic or

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general magnetic nanomaterials, have attracted the great attention of researchers and scientists. (14–18) Spinel ferrite containing $M^{II}Fe_2O_4$ (where $M^{II} = Co^{2+}, Ni^{2+}, Zn^{2+}, Mn^{2+}$, and so on) shows noteworthy physicochemical properties. (19–24) Among spinel ferrites, cobalt spinel ferrite ($CoFe_2O_4$) is important from the biomedical application perspective because of its great saturation magnetization, high permeability, and no favored direction of magnetization. (25) In addition, it displays high anisotropy of the order $190\text{--}280\text{ kJ/m}^3$ in correlation with alternate ferrites. Hence, the magnetic moment of $CoFe_2O_4$ relaxes with a much slower rate than other iron oxide nanoparticles of same size. This suggests that $CoFe_2O_4$ nanoparticles with a nanometric size range can be utilized as an alternative of iron oxide nanoparticles in hyperthermia applications. Utilization of $CoFe_2O_4$ nanoparticles in the biomedical field is very monotonous due to a few issues, for example, aggregation of nanoparticles in solution and the toxicity of the material. To overcome these issues, coating $CoFe_2O_4$ nanoparticles with suitable surfactants, for example, oleic acid, polyethylene glycol, and so forth, produces a protecting layer and lessens the direct and immediate exposure of the $CoFe_2O_4$ nanoparticles surface to the organic climate. Oleic acid (OA) is one of the promising coating agents having complementary anticancerous and anti-inflammatory behavior. (26) Oleic acid coating helps us to stabilize the nanoparticles in fluidic media and enhances the dispersion stability of the nanoparticles with less agglomeration. Also, oleic acid coating increases the biocompatibility of the nanoparticles, which is one of the main concerns of the biomedical applications of nanoparticles. (27)

In recent decades, surface-functionalized spinel ferrites, especially $CoFe_2O_4$ nanoparticles prepared by utilizing wet chemical techniques were explored by the academic and scientific network for biomedical applications, for example, directed drug loading, magnetic hyperthermia, and magnetic resonance imaging. (28,29) These analyses were fundamentally centered on the inductive heating properties of $CoFe_2O_4$ nanoparticles synthesized by the hydrothermal method and the sol–gel autoburning technique. However, very few reports are available on water-dispersible $CoFe_2O_4$ nanoparticles prepared by the chemical co-precipitation method and functionalized by oleic acid. The $CoFe_2O_4$ nanoparticles are effectively dispersed and stable in combination with water and ethylene glycol in contrast to other fluidic mediums, which results in a superior self-warming at lower attractive field. In correlation with other synthesis routes, the chemical co-precipitation strategy showed numerous advantages, for example, better homogeneity, small particle size, moderately low reaction temperature, thin size distribution, low cost, and so on. (30,31) Thus, herein, we report the synthesis, characterization, and induction heating evaluation of surface-modified $CoFe_2O_4$ nanoparticles co-dispersed in water/ethylene glycol. Oleic acid-coated cobalt ferrite nanoparticles were synthesized using the chemical co-precipitation route and characterized by standard techniques for structure, morphology, and magnetic properties analysis. Further, the characterized surface-modified $CoFe_2O_4$ was then added in ethylene glycol/water with various concentrations and characterized by the induction heating technique for

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The pristine and surface-functionalized CoFe₂O₄ samples were characterized by XRD and SAED, respectively. The recorded X-ray diffraction (XRD) patterns of PR-CF and SM-CF are shown in Figure 1, which reveals that both the PR-CF and SM-CF samples exhibit good crystalline nature that confirms the formation of a pure-phase nanoparticle without any impurity phase. Rietveld refinement of both the PR-CF and SM-CF samples was done by FullProf Suite software. It is confirmed from Figure 1 that functionalization of the oleic acid on the surface of the CoFe₂O₄ nanoparticles did not modify the crystal structure of the PR-CF. A small noise was recorded in the XRD pattern of SM-CF due to the amorphous nature of oleic acid layer on the surface of the CoFe₂O₄ nanoparticles. Table 1 shows the values of structural parameters of PR-CF and SM-CF nanoparticles deduced from XRD data.

Figure 1

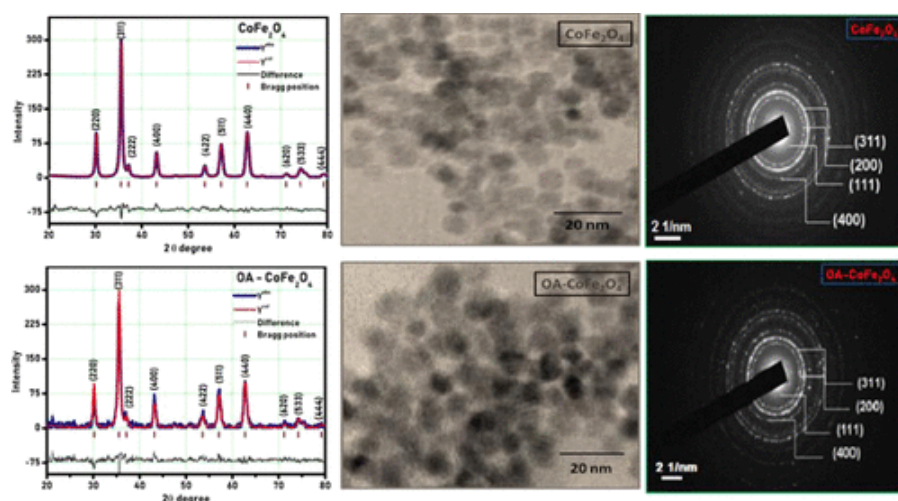


Figure 1. XRD patterns, transmission electron microscopy (TEM) images, and selected area electron diffraction (SAED) patterns of PR-CF and SM-CF nanoparticles.

Table 1. Values of Lattice Constant (a), Crystallite Size (D), Particle Size (t), X-ray Density (d_X), Bulk Density (d_B), and Porosity (P) of PR-CF and SM-CF Nanoparticles

sample	a (Å)	D (nm)	t (nm)	d_X (g/cm ³)	d_B (g/cm ³)	P (%)
PR-CF	8.355	9.64	9.5	5.336	3.633	33.53
SM-CF	8.362	10.40	10	5.364	3.668	31.83

The morphology of PR-CF and SM-CF analyzed by transmission electron microscopy (TEM) as shown

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pattern of SM-CF confirmed the functionalization of oleic acid on the surface of PR-CF, which did not modify the crystal structure of the PR-CF. The brightness of the SAED pattern slightly decreased in the case of SM-CF, due to the amorphous nature of the oleic acid layer on the surface of CoFe_2O_4 .

Thermogravimetric analysis (TGA) of PR-CF and SM-CF was studied to examine the phase development temperature of the spinel cubic structure. The TGA plot of PR-CF and SM-CF is shown in Figure 2. The curve shows the elimination of nitrates trailed by the development of the spinel ferrite phase with respect to temperature. The TGA curve demonstrates total weight losses of 21.18 and 24.87% for the PR-CF and SM-CF samples, respectively, as seen in Figure 2. Also, it is noted from the TGA curve that there is no notable weight loss beyond 450 °C, which is accredited to spinel ferrite phase development. The decomposition of the SM-CF sample started at 170 °C and ended at 440 °C with a total weight loss of 24.87%. This weight loss of 24.87% emerged from the disintegration of oleic acid, which is presented on the core of the SM-CF sample. This validates the total % of oleic acid as 24.87% on the SM-CF core.

Figure 2

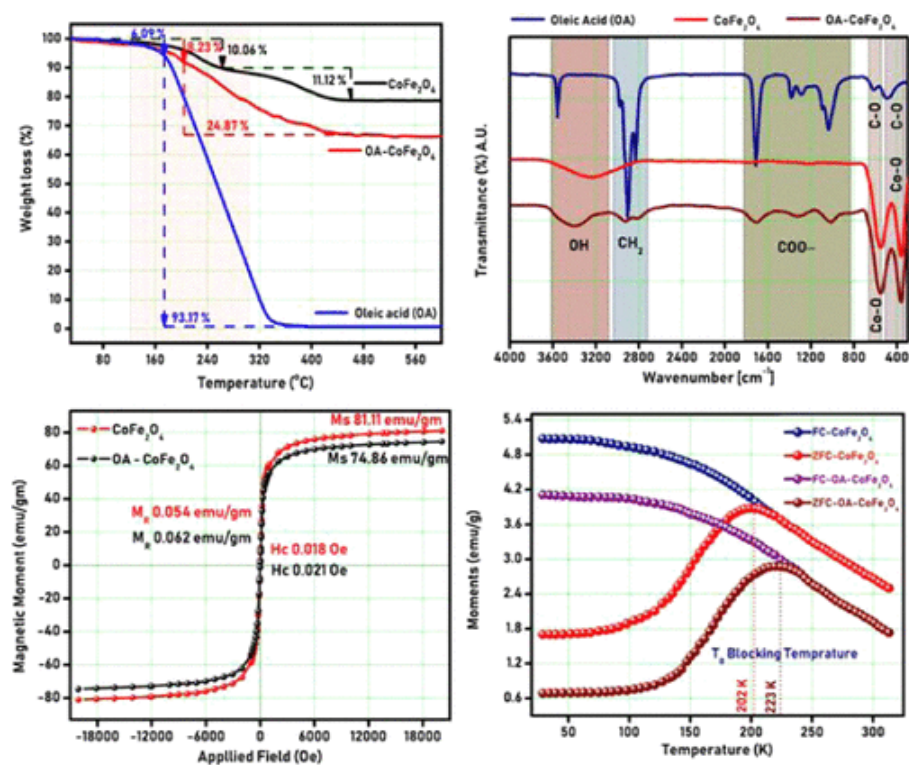


Figure 2. TGA plot, Fourier transform infrared (FT-IR) spectra, M–H plot, and field-cooled and zero-field-cooled (FC–ZFC) plot of PR-CF and SM-CF nanoparticles.

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conformation. The peak at 3026 cm^{-1} is due to the asymmetric stretching linked with the CH_2 band. This observation indicates the existence of carboxylic molecules on the nanoparticle surface, which also confirms the functionalization of the oleic acid layer over the cobalt ferrite nanoparticles. The obtained peaks are well matched with earlier reports. (34)

The magnetic properties of PR-CF and SM-CF were studied by M–H plots with an applied field of $\pm 21\text{ kOe}$ at 300 K , and the obtained results are shown in Figure 2. From Figure 2, it is revealed that the saturation magnetization (M_S) value of SM-CF (76.86 emu/g) is slightly less in comparison to M_S values of PR-CF (81.11 emu/g). The slight decrease in the M_S value for SM-CF is due to the oleic acid layer, which decreases the interparticle interface and the exchange coupling energy, which in turn reduces the magnetization. Both the PR-CF and SM-CF samples show very small and negligible values of remanent magnetism (M_R) and coercivity (H_C), which confirms the superparamagnetic nature of both the samples. (35) The decrease of the magnetization values can also be linked with the mass correction of the nonmagnetic oleic acid content covered on the core of the SM-CF sample. The mass correction can be evidenced by thermogravimetric analysis, which ensured 24.87% oleic acid content over the core of the SM-CF sample. The field-cooled and zero-field-cooled (FC–ZFC) plots of PR-CF and SM-CF are shown in Figure 2. The FC–ZFC plots show blocking temperature (T_B) of PR-CF and SM-CF. The ZFC curve reached the maximum at about 202 K for PR-CF and 223 K for SM-CF, which correspond to the blocking temperature (T_B) of the sample. Above T_B , the sample shows superparamagnetic behavior. The blocking temperature (T_B) of SM-CF was slightly greater compared to PR-CF, which is caused due to oleic acid functionalization.

The heat generation ability of both the PR-CF and SM-CF samples was tested using an induction heating system. Both the PR-CF and SM-CF samples were dispersed in ethylene glycol/water with various concentrations and characterized by the induction heating technique for evaluation of their self-heating characteristics. A series of temperature versus time measurements were done by varying the ethylene glycol/water (EG/W) proportion for better understanding of the self-heating characteristics of the prepared CoFe_2O_4 nanoparticles. Figures 3 and 4 show the hyperthermia curves for different nanoparticle concentrations as well as varying ethylene glycol/water proportions for PR-CF and SM-CF samples, respectively.

Figure 3

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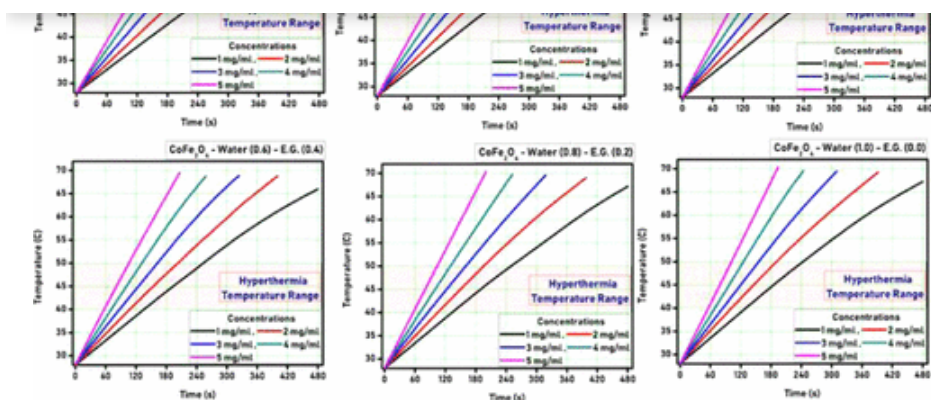


Figure 3. Hyperthermia curves for PR-CF nanoparticles.

Figure 4

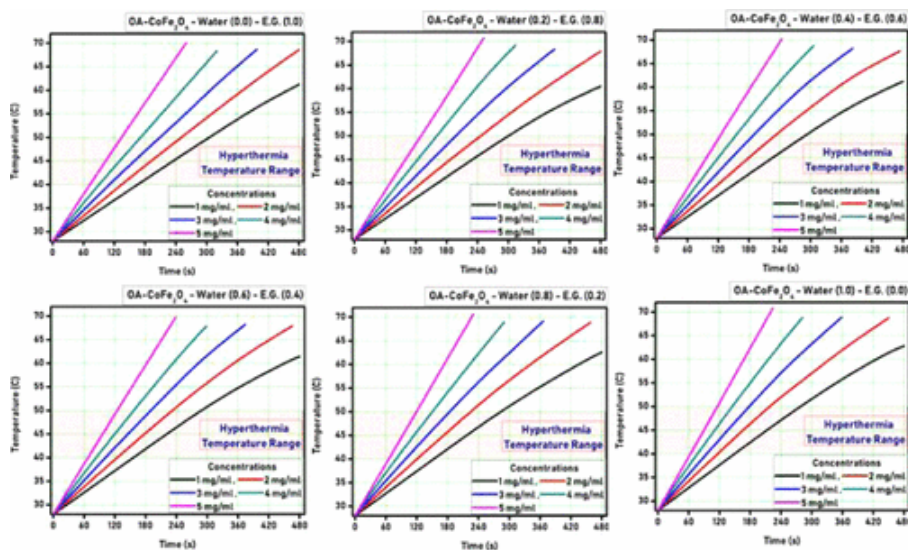


Figure 4. Hyperthermia curves for SM-CF nanoparticles.

It is observed from [Figures 3](#) and [4](#) that all of the curves show a gradual increase in temperature with respect to time. The time required for the temperature rise decreased with the increase of nanoparticle concentration for both the PR-CF and SM-CF samples. The hyperthermia temperature was achieved at 65 s by the PR-CF sample with 1 mg/mL concentration, whereas for 5 mg/mL concentration, it was 55 s. Similarly, the hyperthermia temperature range was achieved at 70 s by the SM-CF sample with 1 mg/mL concentration, whereas for 5 mg/mL concentration, it was 60 s. It is also observed from the curves that the increase in temperature was slightly decreased after the oleic acid coating over the CoFe_2O_4 .

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Figure 5 shows the SAR plots for different nanoparticle concentrations as well as varying ethylene glycol/water proportion for PR-CF and SM-CF samples, respectively. It is observed from Figure 5 that the SAR value decreases with an increase in the nanoparticle concentration, whereas it increases with an increase in water concentration. The highest SAR values of the order of 248 W/g was observed for the PR-CF sample with 1 mg/mL concentration. This SAR value was decreased to 216 W/g for the SM-CF sample due to oleic acid coating. This shows that the SM-CF sample with 1 mg/mL concentration is sufficient to produce the required heat for hyperthermia treatment.

Figure 5

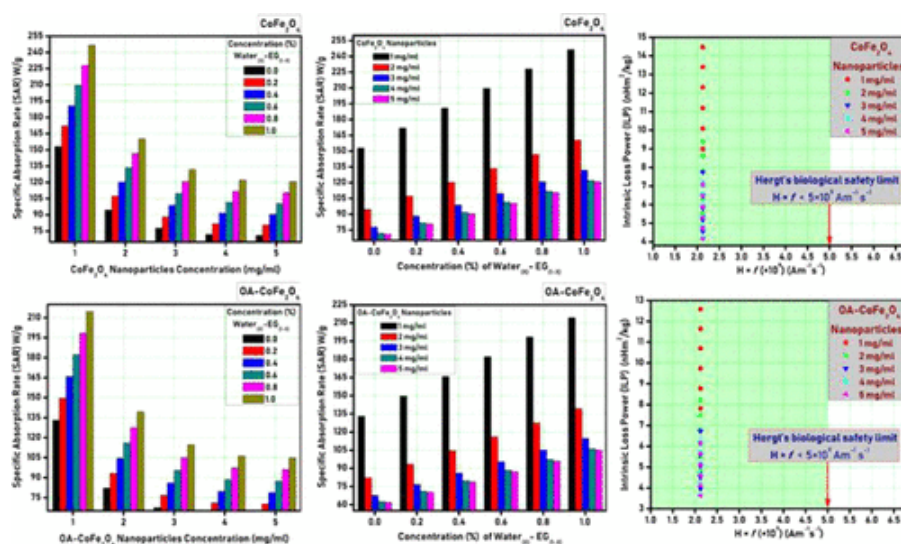


Figure 5. SAR and ILP plots for PR-CF and SM-CF nanoparticles.

3. Conclusions

[Jump To](#)

Oleic acid-coated CoFe_2O_4 nanoparticles were successfully synthesized using the chemical co-precipitation route. The Rietveld refined XRD pattern of CoFe_2O_4 nanoparticles indicated the formation of a cubic-spinel single-phase structure with the $Fd\bar{3}m$ space group. The average crystallite size (~ 12 nm) confirmed the nanocrystalline appearance of the prepared CoFe_2O_4 nanoparticles. TEM images revealed the spherical nature of both (CoFe_2O_4) and ($\text{OA-CoFe}_2\text{O}_4$) samples. The absorption bands in the FT-IR spectrum at $\sim 3418, 3026, 1628, 1404, 1068, 845, 544,$ and 363 cm^{-1} affirmed the spinel ferrite formation and OA attachment. The $M-H$ curve recorded at room temperature showed the

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CoFe₂O₄ nanoparticles were synthesized by the chemical co-precipitation route. Nitrates of cobalt and iron were independently dissolved in a stoichiometric ratio of 1:2 to get the precursors. The pH of the mixed solution was noted as 3. The pH was then adjusted by adding 2 M NaOH. A black precipitate was obtained by heating the solution up to boiling temperature for 2 h. The obtained precipitate was washed several times with water. Furthermore, 2 M nitric acid (HNO₃) was added to the precipitate to eliminate impurities. The nanoparticles were filtered and dried at 55 °C for 24 h. The surface of the CoFe₂O₄ nanoparticles was modified with oleic acid by the ultrasonication process. The pristine and surface-modified CoFe₂O₄ samples were coded as PR-CF and SM-CF, respectively. The characterization techniques used in the current work are summarized in [Table 2](#).

Table 2. Characterization Techniques Employed in the Current Work

characterization tool	make and model	specification
"XRD"	Make: Rigaku; model: Ultima IV	room temperature; 2θ rang
"FT-IR"	Make: Thermo Scientific; model: Nicolet iS10	room temperature; wavenu
"TGA"	Make: Shimadzu; model: TGA-51	nitrogen atmosphere, room
"TEM"	Make: PHILIPS; model: CM-200	energy: 20–200 kV; resolut
"VSM"	Make: "Lakeshore" model: VSM 7407	room temperature and 5 K;
"induction heating"	Make: Ambrell model: Easy Heat 8310	field: 35 kA/m frequency: 3

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[Jump To](#)

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The authors declare no competing financial interest.

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P.B.K. acknowledges Dr. Raghmani S. Ningthoujam, Scientific Officer F, Bhabha Atomic Research Centre, Mumbai, for providing induction heating measurement facility. S.B.S. acknowledges Department of Science and Technology (DST), Govt. of India, for DST-INSPIRE Fellowship (IF170288).

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