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Combustion synthesized Fe doped CeO₂ powder-characterization, optical absorption and EPR spectroscopy

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Abstract Fe doped CeO₂ powder was prepared using solution combustion method. Powder X-ray diffraction and scanning electron microscopy methods are used to characterize the combustion derived powder. The optical absorption spectrum exhibits three bands due to Fe³⁺ and Fe²⁺ ions. Electron paramagnetic resonance (EPR) spectrum of this sample exhibits number of resonance signals due to Fe³⁺ ions. The number of spins (N) participating in resonance and its paramagnetic susceptibility (χ) has been evaluated. From EPR and optical studies it is observed that iron ions are present in trivalent state.

1 Introduction

Among the various rare-earth based oxide materials CeO₂ is the most promising rare earth oxide which has been the popular subject of research. Since CeO₂ endows with some exceptional features such as a thinner interfacial layer,

higher capacitance value, high dielectric constant, high effective electric field across the tunnelling oxide and lower leakage current, therefore it is being used extensively in semiconductor fabrication over the past few years [1–5]. CeO₂ based rare earth oxide ceramics have wide range of applications in the field of laser glass, integral optical instruments, fluorescent lamps and so on [2–8]. CeO₂ has been studied under the photonic band gap materials and has become a popular subject of interests in the research area.

There are several researches being carried out based on the preparations and properties of heterogeneous photo catalyst CeO₂ coupled with other materials throughout last decade [8–10]. CeO₂ has been proved as an excellent photo catalyst in the modern catalytic industry because of its high photocatalytic activity, low cost and environment friendly behaviour [11–13]. As compared to all other stabilized zirconia system, CeO₂ stabilized zirconia is considered to have an excellent thermal stability and high toughness. Ceria derived oxides, especially Zr doped cerium oxides have drawn the significant attention of researchers and intensive studies reported on them for past several years [14–16]. An intensive study of electrical conductivity and defect structure of CeO₂–ZrO₂ mixed oxide has been done by Lee et al. [17]. Wang et al. [18] have reported the preparation of inverse opal cerium dioxide for optical properties research. Luisetto et al. [19] have investigated the catalytic performances of Ni/CeO₂–Al₂O₃ catalysts for the dry reforming of methane. Due to some peculiar redox properties and variable valence states (4+ and 3+), ceria and ceria derived oxides play an important role as a heterogeneous catalyst in the field of industrial applications such as water gas shift reactions, solid oxide fuel cells anodes, automotive three way exhaust, etc. [20–23].

It was found that photocatalytic properties of CeO₂ nanomaterials can be improved further by doping with

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transition metal (Fe), which enhances the oxygen storage capacity of ceria lattice [24]. Fe doped CeO₂ nano particles have emerged as efficient photocatalysts in the visible region for dye waste water treatment and for pigmented ultraviolet filter applications [25]. Tiebao et al. [26] have investigated the fabrication of nano CeO₂ and application of nano CeO₂ in Fe matrix composites. Influence of Fe³⁺ doping on optical properties of CeO₂ nanopowders, has been discussed by Radović et al. [27]. Wang et al. [28] have reported the enhanced photocatalytic performance of ordered mesoporous Fe doped CeO₂ catalysts for the reduction of CO₂ with H₂O under simulated solar irradiation. From the above several reports, it appears that cerium oxides have different potential applications and been used as catalysts, phosphors, polishing materials, fuel cells, gas sensors and so on in various technological fields. It is well known that, among the different valence states of Fe, the trivalent state is the most stable one. Fe³⁺ ions have significant use as active media in solid state optical materials/display/lasers devices etc. These ions interact well with the host lattice and therefore are widely investigated for numerous optical spectroscopic properties. Furthermore, Fe³⁺ ion is interesting because of its extreme sensitivity towards optical and electron paramagnetic resonance (EPR) properties and their dependency on the nature of the host matrix. Although there are plenty of literature reports available on the doped and un-doped CeO₂, there are only some reports on Fe doped CeO₂ materials.

Further, CeO₂ is being used in phosphor systems to improve the luminescence properties of material by either mixing or coating. However, CeO₂ as a host for the Fe ions doping has not been often reported. In view of this, in this work, we have selected CeO₂ as a host material for the transition metal ions. The addition of transition metal ions to the inorganic oxide material changes them to exhibit the structural, chemical, and physical behavior. Among the transition metal oxides, iron is of much interest in the contemporary and emerging technology, wherein it finds applications in the fields like microelectronics, solid state ionics etc. It is noticed that electron paramagnetic resonance (EPR) is used as a tool for the characterization of rare earth and transition metal ions in optical and laser crystals, because of their relevance in optoelectronic devices. EPR spectra of Fe³⁺ ions in various glasses have been extensively recorded and studied [29, 30]. Accordingly, Fe³⁺ is a very useful probe to obtain the information about local environment in the host lattice [31–33]. To the best of our knowledge, no details regarding the optical properties of Fe doped CeO₂ have been reported in the literature. It is widely accepted that the EPR is an amenable method for elucidating the ground state properties of transition metal ions. In this context, we have studied the

Fe doped CeO₂ using optical and EPR spectroscopic techniques. It will be interesting to understand the site symmetry of Fe when incorporated into CeO₂ matrix.

The Fe doped CeO₂ sample was prepared by combustion route. The advantages of this synthesis method are possibility to prepare good homogeneity through mixing at the molecular level, safe, easy and no requirement of any special arrangements for preparation. In the present work a detailed investigation on cerium oxide doped with iron has been undertaken using spectroscopic methods such as X-ray diffraction (XRD), scanning electron microscopy (SEM), electron paramagnetic resonance (EPR), optical absorption and the results obtained from these studies are discussed.

2 Experimental details

2.1 Synthesis

We prepared sample of Fe doped CeO₂ utilizing the combustion process. In a typical synthesis, 4 g of Ce(NO₃)₃·6H₂O, 0.0744 g of Fe(NO₃)₃·9H₂O and 1.4 g of urea (NH₂CONH₂) was dissolved in a minimum quantity of deionized water in a China dish with 300 ml capacity to obtain a homogeneous solution. The solution was then transferred into a furnace preheated to 550 °C. The water quickly evaporated and the mixture formed foam, within which a vigorous reaction between the nitrates and urea soon initiated and ended. The entire combustion process was completed in about 3–5 min. The resultant fluffy masses were crushed into a fine powder. Then the powder was transferred to a 50 ml alumina crucible to be heat-treated at 1000 °C for 2 h in air to remove by-products and reduce internal strains before it was used for further characterization.

2.2 Characterization

Powder XRD pattern was recorded in the 2θ range from 10° to 80° on a Philips X'Pert X-ray diffractometer with graphite monochromatized CuK_α radiation and nickel filter at a scanning step of 0.03°. The morphology of the prepared sample was investigated using SEM on Hitachi S-3200N instrument. The UV-Vis absorption spectrum of sample was measured in diffuse reflectance mode using a Cary 6000i UV-Vis-NIR spectrophotometer equipped with an integrating sphere. A powdered sample of 100 mg was taken in a quartz tube for the EPR measurements. The EPR spectra of sample were recorded on a JEOL FE1X EPR Spectrometer, operating in the X-band frequencies, with a field modulation of 100 kHz.

3 Results and discussion

3.1 X-ray diffraction analysis

XRD measurements were carried out for analyzing crystal structure and its phase. The XRD pattern of combustion derived Fe doped CeO_2 powder has been measured and is shown in Fig. 1. These results show the sample to exhibit face-centered cubic fluorite crystal structure² belonging to space group $Fm\bar{3}m$ (No. 225). The fluorite structure with the crystalline planes of (111), (200), (220), (311), (222), (400), (331) and (420) are matched with that of pure CeO_2 (JCPDS, File No. 34-0394). Figure 1 shows that doping with Fe^{3+} ions has no effect on shapes and position of diffraction peaks. Moreover, there is no additional signature in XRD pattern of Fe doped CeO_2 powders which imply that² the dopant was incorporated into the CeO_2 structure, which indicates the formation of single phased CeO_2 . This XRD analysis indicated that the dopant do not participate in overall crystal formation except replacing of Ce/sitting between few atoms. The average crystallite size of the Fe doped CeO_2 sample was estimated by using Scherrer's method, $d = k\lambda/\beta \cos\theta$, where k is a constant which is equal to 0.89, β is the full width at half maxima in radians, λ is the wavelength of X-ray and θ is the Bragg's angle. The average crystallite size was found to be 48 nm.

3.2 Morphological analysis

SEM study was carried out to investigate the morphology of the synthesized powder. Figure 2 displays higher magnification SEM image of the Fe doped CeO_2 powder. SEM studies showed that, there is a cluster of large number of sphere like balls. There is no uniformity in distribution of particle; as almost all the smaller particles in a cluster look alike. The non-uniform behavior of the particles can be attributed to the non-uniform distribution of temperature and mass flow in the combustion flame.

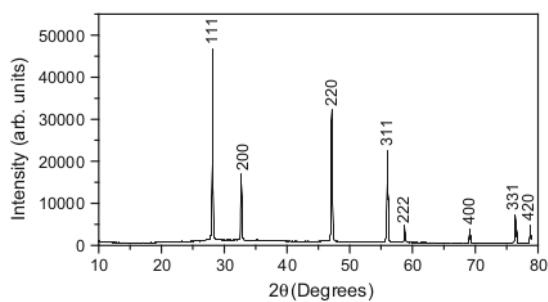


Fig. 1 Powder XRD pattern of $\text{CeO}_2:\text{Fe}^{3+}$

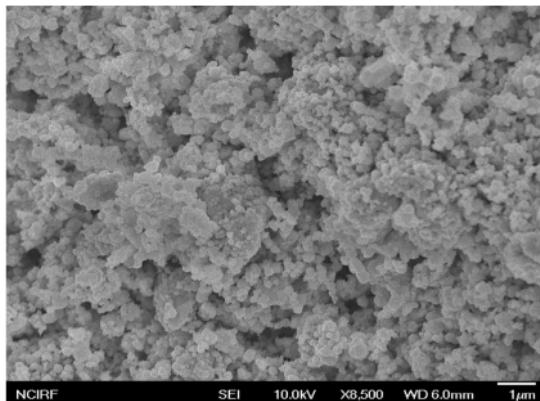


Fig. 2 SEM micrograph of $\text{CeO}_2:\text{Fe}^{3+}$

3.3 Absorption analysis

The absorption spectrum of Fe doped CeO_2 sample is shown in Fig. 3. From the figure it is evident that two broad bands have been observed at around 535 nm ($18,692 \text{ cm}^{-1}$) and 906 nm ($11,037 \text{ cm}^{-1}$). The band at around 535 nm ($18,692 \text{ cm}^{-1}$) has been attributed to Fe^{3+} ions in octahedral symmetry and assigned to the transition $^6\text{A}_{1g}(\text{S}) \rightarrow ^4\text{T}_{2g}(\text{G})$ [32]. The weak band at around 671 nm ($14,903 \text{ cm}^{-1}$) has been attributed to Fe^{3+} ions in octahedral symmetry and assigned to the transition $^6\text{A}_{1g}(\text{S}) \rightarrow ^4\text{T}_{1g}(\text{G})$. The band at around 906 nm ($11,037 \text{ cm}^{-1}$) has been attributed to Fe^{2+} ions and assigned to the transition $^5\text{E}(\text{D}) \rightarrow ^5\text{T}_{2}(\text{D})$ in tetrahedral symmetry [33]. For Fe^{3+} ions, spin-forbidden transitions are allowed. Therefore the intensity of the bands should be low. In the present case, two or more bands due to Fe^{3+} ions were merged into one band and resulted a broad band at around 535 nm ($18,692 \text{ cm}^{-1}$). For Fe^{2+} ions, spin-allowed transitions are possible. Therefore a broad band is resulted at around 906 nm ($11,037 \text{ cm}^{-1}$). For iron 2+, 3+, 4+ even higher oxidation

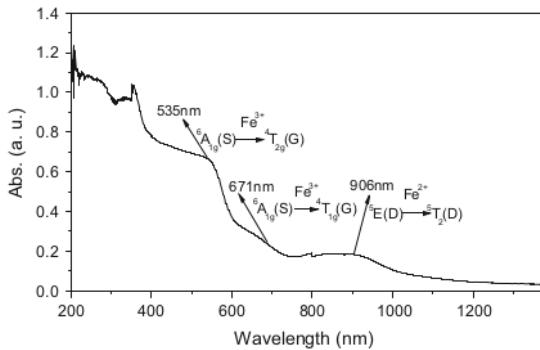


Fig. 3 Absorption spectrum of $\text{CeO}_2:\text{Fe}^{3+}$

states are found to occur. Among these 3+ oxidation state is more stable for iron. But Fe^{2+} and Fe^{3+} are optically active in this sample. During sample preparation, some of the Fe^{3+} ions are converted into Fe^{2+} ions. In general, experimental data cannot be interpreted correctly in terms of 3+ oxidation state alone, but have to be analysed in view of the coexistence of Fe ions of different vacancies. In the next section, Fe^{2+} ions were not observed in EPR studies. Mehdikhani and Borhani [34] studied optical spectroscopy of sodium silicate glasses prepared with nano and micro sized iron oxide particles. They observed three bands at around 380, 420, 435 nm due to Fe^{3+} ions. They also observed a broad band at around 1050 nm due to Fe^{2+} ions. Klement et al. [35] studied spectroscopic analysis of iron doped glasses. They observed two bands at around 5000 and 10,000 cm^{-1} . These bands were due to Fe^{2+} ions and assigned to the transitions $^5\text{T}_2(\text{D}) \rightarrow ^5\text{E}(\text{D})$ and $^5\text{E}(\text{D}) \rightarrow ^5\text{T}_2(\text{D})$ in octahedral and tetrahedral symmetries, respectively. In view of the above information the band at around 906 nm is most likely due to Fe^{2+} ions. Singh et al. [36] studied optical absorption studies of Fe_2O_3 and CeO_2 doped glasses. They observed two weak bands at around 408 and 424 nm due to Fe^{3+} ions in octahedral symmetry. They also observed two bands at around 411 and 431 nm due to excitons for CeO_2 doped glasses. The bands located between 200 and 350 nm are the host absorption bands due to charge transfer (CT) from O^{2-} to Ce^{4+} , because of the energy gap between $\text{O}(2p)$ band and $\text{Ce}(4f)$ is in the range of 6–8 eV [37, 38].

3.4 EPR studies

EPR spectrum of un-doped does not show any absorption signals. This indicates that the starting materials do not contain any paramagnetic impurities and defects. When Fe is added to the CeO_2 as dopant, then this exhibits resonance signals due to Fe^{3+} ions. Figure 4a shows EPR spectrum of $\text{CeO}_2:\text{Fe}^{3+}$ at RT. This spectrum exhibits the effective g values at $g = 7.77, 4.05, 2.07$ and 1.93 . Figure 4b shows the EPR spectrum of $\text{CeO}_2:\text{Fe}^{3+}$ at 110 K. This spectrum gives the effective g values at $g = 24.92, 7.79, 4.17, 2.71, 2.21$ and 2.01 . These large g values are present when certain symmetry elements [3] are present. The large g values are usually expressed by spin-Hamiltonian of the form [39]

$$\mathbf{H}_s = g\beta\mathbf{HS} + D[S_z^2 - (1/3)S(S+1)] + E(S_x^2 - S_y^2) \quad (1)$$

where the terms involving D and E are the axial and orthorhombic crystal-field components respectively, S is the spin of 20 tor and g is the spectroscopic splitting factor. Free Fe^{3+} ions belong to the d^5 configuration with $^6\text{S}_{5/2}$ as the ground state, without any spin-orbit interaction. The g value of free Fe^{3+} ions is expected to lie very close to the free-electron value of 2.0023. The experimental g values of

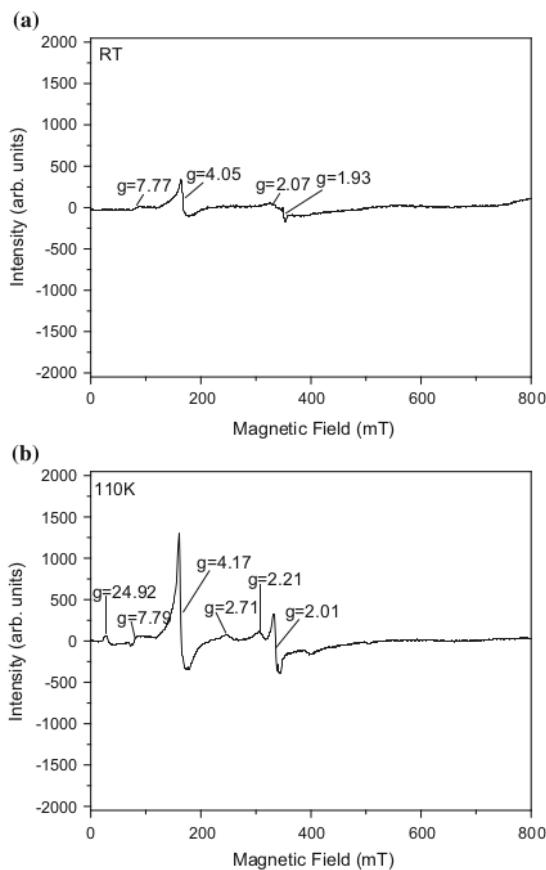


Fig. 4 The EPR spectrum of $\text{CeO}_2:\text{Fe}^{3+}$ at **a** room temperature and **b** 110 K

Fe^{3+} species in poly crystalline solids often deviate significantly from 2.0023. When Fe^{3+} ions are placed in a ligand field environment, the $^6\text{S}_{5/2}$ ground state splits into three Kramers' doublets $|\pm 1/2\rangle$, $|\pm 3/2\rangle$ and $|\pm 5/2\rangle$. The resonance signal at $g = \sim 4.05, 4.17$ arises from the middle Kramers' doublet $|\pm 3/2\rangle$ for Fe^{3+} ions in almost completely rhombic environments with the ratio of the zero-field splitting parameters $|E|/|D| \approx 1/3$ [40, 41]. The g values close to $g \sim 2.0$ is due to Fe^{3+} ions in octahedral symmetry. The large number of g values obtained in the present case at RT and 110 K. It is most likely due to anisotropic nature of Fe^{3+} ions in CeO_2 host. The $g \sim 2.0$ resonance signal may be attributed to either Fe^{3+} ions interacting by dipole-dipole interaction in distorted octahedral field or to their super exchange interactions within the pairs of Fe ions [42, 43]. These large number of g values indicate that the iron ions are in trivalent state and the site symmetry is distorted octahedral. In the present case, EPR signal is not observed for Fe^{2+} ions. It is known

that in the case of iron, both (2+ and 3+) redox states can potentially be EPR active, although the 2+ state normally is not. Sivaramaiah et al. [44] studied EPR studies of Fe³⁺ ions in amethyst. They observed two broad signals at g = 10.8, 4.0 and one sharp signal at g = 2.002. The broad signals have been attributed to Fe³⁺ clusters on surfaces. The sharp signal has been attributed to oxygen vacancy [22] electron center E_{1'}. Sivaramaiah and Pan [45] studied thermodynamic and magnetic properties of surface Fe³⁺ ions on quartz. They observed three resonance signals at g = 2.00, 4.28 and 15. The resonance signal at g = 4.28 is attributed to the substitutional Fe³⁺ ions at the Si site. The signal at g = 2.00 has been attributed to oxygen vacancy center E_{1'}. The signal at g = 15 has been attributed to Fe³⁺ clusters. The remarkable feature observed for this sample is that the optical spectrum observed due to Fe³⁺ as well as Fe²⁺ whereas EPR signal is due to Fe³⁺ only.

3.4.1 Absolute number of spins

The absolute number of spins (N) participating in resonance can be calculated by comparing the area under the absorption curve with that of a standard of known concentration. Weil and Bolton [46] gave the following formula including the experimental parameters of both the sample and the standard

$$N = \frac{A_x(\text{scan}_x)^2 G_{\text{std}}(B_m)_{\text{std}}(g_{\text{std}})^2 [S(S+1)]_{\text{std}}(N_{\text{std}})}{A_{\text{std}}(\text{scan}_{\text{std}})^2 G_x(B_m)_x(g_x)^2 [S(S+1)]_x}, \quad (2)$$

where A is the area under the absorption curve, which is obtained from double integration of the first derivative spectrum, scan is the magnetic field corresponding to unit length of the spectrum, G is the gain, B_m is the modulation amplitude, g is the g factor, S is the spin of the system in its ground state (S = 5/2 and 1/2 for Fe³⁺ and Cu²⁺, respectively). N_{std} denotes the number of spins in 100 mg of CuSO₄·5H₂O standard. The absolute number of spins for the Fe³⁺ signal at g = 4.05, 4.17 in CeO₂ was found to be order of 10¹⁹ spins/m³ at RT and 110 K. This spin order is consistent with the spin order reported in the literature [36].

3.4.2 Gibbs free energy

The Gibbs free energy (ΔG) can be calculated using the following equation [44, 45]

Table 1 The g values, number of spins (N), Gibbs free energy (ΔG), magnetic susceptibility (χ), Curie constant (C) and effective magnetic moment (μ_{eff}) for CeO₂:Fe³⁺ at RT and 110 K

T (K)	g value	N (spins/m ³)	ΔG (kJ/mol)	χ (m ³ /kg)	C (emu/mol)	μ_{eff} (μ_B)
RT	4.05	20.59×10^{19}	-43.16	20.46×10^{-5}	0.061	11.9
110	4.17	97.29×10^{19}	-16.34	10.25×10^{-4}	0.30	12.18

$$\Delta G = 2.303 RT \log_{10}(k_B T / \lambda h) \quad (3)$$

Here ' ΔG ' is the Gibbs free energy, 'R' is the universal gas constant, ' k_B ' is the Boltzmann constant, 'T' is the absolute temperature, ' λ ' is the rate constant [22] is equal to the absolute number of spins per m³, and 'h' is Planck's constant. The Gibbs free energy of CeO₂:Fe³⁺ is found to be -43.16 and -16.34 kJ/mol at RT and 110 K, respectively. Thus the stability of this sample is high at RT as Gibbs free energy is low at RT. Lower the Gibbs free energy value, higher the stability. The negative sign indicates that exothermic reaction takes place between the dopant and the host lattice and there is strong attraction between the dopant and the lattice.

3.4.3 Magnetic susceptibility

The magnetic susceptibility χ for the g = 4.05, 4.17 signals can be determined using the following expression [47, 48]

$$\chi = \frac{(Ng^2\beta^2 J(J+1))}{18} / 3k_B T, \quad (4)$$

where N is the number [18] of spins per m³, g is the g factor and is equal to 4.05, 4.17, β is the Bohr magneton, S is the spin quantum number of unpaired electron = 5/2, k_B is Boltzmann constant and T is absolute temperature. Using the above equation, the magnetic susceptibility of the sample at RT was found to be 20.46×10^{-5} m³/kg. Its value at 110 K was found to be 10.25×10^{-4} m³/kg. The calculated magnetic susceptibility values are consistent with the values reported in the literature [44, 45]. The advantage of this EPR method is that the diamagnetic contribution of magnetic susceptibility can be eliminated. For paramagnetic contribution of the sample, the magnetic susceptibility is calculated.

3.4.4 Curie constant

The empirical Curie constant can be found using the following expression [47, 48]

$$C = \frac{Ng^2\beta^2 J(J+1)}{3k_B}, \quad (5)$$

where N is number of spins/m³, g = 4.05, 4.17, β is Bohr magneton, J is total angular momentum quantum number, k_B is Boltzmann constant. The Curie constant was found to

be 0.061 and 0.30 emu/mol at RT and 110 K, respectively. The Curie constant has increased with the decrease of temperature which demonstrates that the paramagnetic interactions increase with the decrease of temperature. The Curie constant indicates the nature of magnetic interactions and the low value of this constant is attributed to high antiferromagnetic interactions. It may also be attributed to the formation of Fe^{3+} - Fe^{3+} ions pairs, if the exchange coupling between these ions is antiferromagnetic in nature, which is expected to result in a reduction of the Curie constant [49].

3.4.5 Experimental effective magnetic moment

The experimental effective magnetic moment can be determined using the following expression [50]

$$\mu_{\text{eff}} = \frac{\sqrt{3k_B C}}{\sqrt{N}}, \quad (6)$$

where k_B is Boltzmann constant, C is Curie constant, N is number of spins/ m^3 . The theoretical magnetic moment value for free Fe^{3+} is 5.92 μ_B . The experimental values were estimated to be 11.9 and 12.18 μ_B at RT and 110 K, respectively. The high value at both the temperatures indicates that ferromagnetic interactions are dominant in this sample. The high value also indicates that clusters of high spin Fe^{3+} are found in this sample. Table 1 shows the g values, number of spins (N), Gibbs free energy (ΔG), magnetic susceptibility (χ), Curie constant (C) and effective magnetic moment (μ_{eff}) for CeO_2 : Fe^{3+} powder at RT and 110 K.

4 Conclusions

Fe doped CeO_2 was successfully prepared by the combustion synthesis method. The XRD revealed the presence of single phase of CeO_2 . Scanning electron microscope images of prepared powders showed an agglomeration. Absorption spectrum studies on the Fe doped CeO_2 sample show two main broad absorption bands observed at around 535 nm and 906 nm which is the characteristic of Fe^{3+} and Fe^{2+} ions in octahedral and tetrahedral symmetries, respectively. The weak band centered at around 671 nm is due to Fe^{3+} ions in octahedral symmetry. EPR spectrum of Fe doped CeO_2 sample showed multiple resonance signals having at least 6 'g' values. These resonance signals can be attributed to Fe^{3+} ions. The number of g values is attributed to anisotropic nature of this sample.

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