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CTAB-assisted of Fe_2O_3/CeO_2 nanosized prepared by coprecipitation method

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ABSTRACT: Recently, cerium oxide (CeO_2) nanoparticles have been widely used in engineering and medical sciences due to the diversity of their applications. Fe-Ce nanoparticles were synthesized by simple co-precipitation method via iron nitrate ($Fe(NO_3)_3.9H_2O$) and cerium nitrate ($Ce(NO_3)_3.6H_2O$) as precursor in the presence of cetyltrimethylammonium bromide (CTAB) surfactant. The samples were characterized by high resolution transmission electron microscopy (HRTEM), field effect scanning electron microscopy (FESEM), X-ray diffraction (XRD), vibration sampling magnetometer (VSM), and Fourier transform infrared spectroscopy (FTIR) in different temperature. The XRD results showed that Fe-doped CeO_2 was single-phased with a cubic structure. The particle size of as-prepared samples was around 45 nm and for annealed one was around 32 nm in diameter at 500 °C for 3 hours. The TEM studies showed the squared-like shaped nanosized particles with good distribution particle. The sharp peaks in FTIR spectrum determined the element of Fe-Ce nanoparticles. The result of magnetic measurements showed coercive field and saturation magnetism around 1632 G and 0.055 emu/g, respectively for as-prepared samples.

Keywords: *Chemical synthesis; Coprecipitation method; CTAB; Fe_2O_3/CeO_2; Nanocrystals*

INTRODUCTION

Magnetic nanoparticles are of great interest for researchers from a broad range of disciplines, including magnetic fluids, data storage, catalysis, and bioapplications (Farahmandjou, 2016, Farahmandjou and Khalili, 2013, Farahmandjou and Ramazani, 2015, Jurablu, *et al.*, 2015, Farahmandjou and Soflaee, 2015, Farahmandjou and Soflaee, 2015, Shadrokh, *et al.*, 2016, Khodadadi, *et al.*, 2019, Farahmandjou, 2012, Farahmandjou and Golabiyan, 2016, Farahmandjou, 2010, Akhtari, et al, 2018, Akhtari, et al, 2018, Khoshnevisan, 2018, Marami, 2018, Farahmandjou, 2018, Farahmandjou and Khalili, 2013, Jurablu, *et al.*, 2015, Farahamndjou, 2013, Farahmandjou, *et al.*, 2016, Jafari, *et al.*, 2018) In the recent years, cerium dioxide (CeO_2 or ceria) has

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received considerable attention because this material shows promising applications in solid oxide fuel cells, environmental catalysis, redox catalysis, and wet catalytic oxidation of organic pollutants (Liu and Flytzani, 1996). However, the band gap of CeO_2 (3.22 eV) has limited the activation of solar energy; only UV light can be applied to generate electron-hole pairs at the beginning of photocatalytic processes (Zarinkamar, *et al.*, 2016, Zarinkamar, *et al.*, 2016). Thus, it is necessary to extend the absorbance of CeO_2 into visible region and reduce the electron-hole pairs recombination. There are many methods to modify light absorption properties of CeO_2, such as metal doping, surface sensitization and coupling with semiconductor that has smaller band gap (Gr¨atzel, 2001). Recently, transition metal doping/loading has been widely used to enhance the light absorption of CeO_2 by our group (Avila, *et al.*, 2010). It has been reported in many works of literature that the metal ions of Fe (Farahmandjou and Dastpak, 2018, Dastpak, *et al.*, 2016), could improve CeO_2 photocatalytic activity towards the visible-light region. Among these metals, Fe has been considered as a candidate owing to its special Fenton reaction of iron. The Fenton process can improve the photocatalytic activity by producing the hydroxyl radicals (OH·) which are very powerful oxidizer in photocatalytic process (Ara, *et al.*, 2001). There are many methods to prepare unloaded CeO_2 and Fe-doped/-loaded CeO_2 nanoparticles such as sol-gel (Yan, *et al.*, 2006), surfactant-assisted precipitation (Terribile, *et al.*, 1998). In our present investigation, FeCe magnetic nano-particles were synthesized using iron nitrate and cerium nitrate precursors. Structural and surface morphological properties are discussed by XRD, HRTEM, FE-SEM, VSM and FTIR analyses.

EXPERIMENTAL DETAIL

Iron-cerium nanoparticles were synthesized by a simple synthesis according to the following manner. Firstly, 1.7 g CTAB surfactant was dissolved in 50 mL pure water and then 3 g $Ce(NO_3)_3.6H_2O$ was added to the solution with stirring at room temperature. After 10 min, 5 mL ethanol was slowly added to the milky-colored solution. Then 3.3 g of $Fe(NO_3)_3.9H_2O$ was added to the solution and synthesis temperature was increased to 90°C. The color of solution changed from milky color to red color by adding iron precursor. The pH=4 was maintained during the synthesis. The product were evaporated for 2 hours, cooled to room temperature and finally calcined at 500°C for 4 hours. All analyses were done for samples without any washing and purification.

The specification of the size, structure and optical properties of the as-synthesis and annealed nanoparticles were carried out. X-ray diffractometer (XRD) was used to identify the crystalline phase and to estimate the crystalline size. The XRD pattern were recorded with 2θ in the range of 4-85° with type X-Pert Pro MPD, Cu-Kα: λ = 1.54 Å. The morphology was characterized by field emission scanning electron microscopy (SEM) with type KYKY-EM3200, 25 kV and transmission electron microscopy (TEM) with type Zeiss EM-900, 80 kV. Fourier transforms infrared spectroscopy (FTIR) with WQF 510. Magnetic measurements were carried out using vibration sampling magnetometer with type VSM 7400 Lake Shore. All the measurements were carried out at room temperature.

RRSULTS AND DOSCUSSION

X-ray diffractometer (XRD) with CuKα radiation, operated at 40 kV, 250 mA was used to identify crystalline phases and to estimate the crystalline sizes. Fig. 1 shows the X-ray diffraction patterns of as-

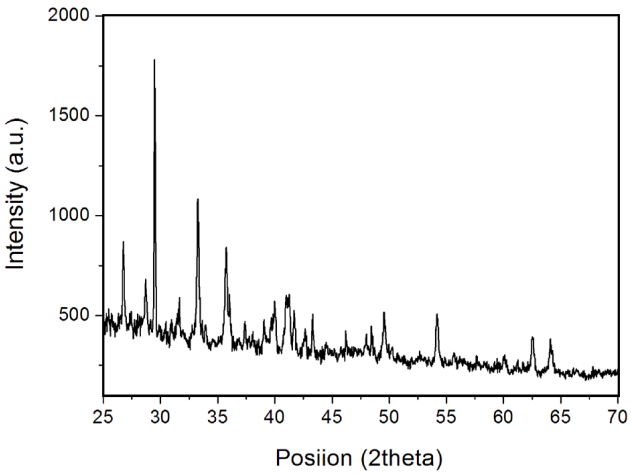

Fig. 1. XRD patterns of as-prepared FeCe sample.

prepared sample. The exhibited peaks correspond the cubic structure. The mean size of the ordered FeCe nanoparticles has been estimated from full width at half maximum (FWHM) and Debye-Scherrer formula (Scherrer, 1918) according to equation the following:

$$D = \frac{0.89\lambda}{B\cos\theta} \quad (1)$$

where, 0.89 is the shape factor, λ is the x-ray wavelength, B is the line broadening at half the maximum intensity (FWHM) in radians, and θ is the Bragg angle. The mean size of as-prepared samples was around 45 nm from this Debye-Sherrer equation.

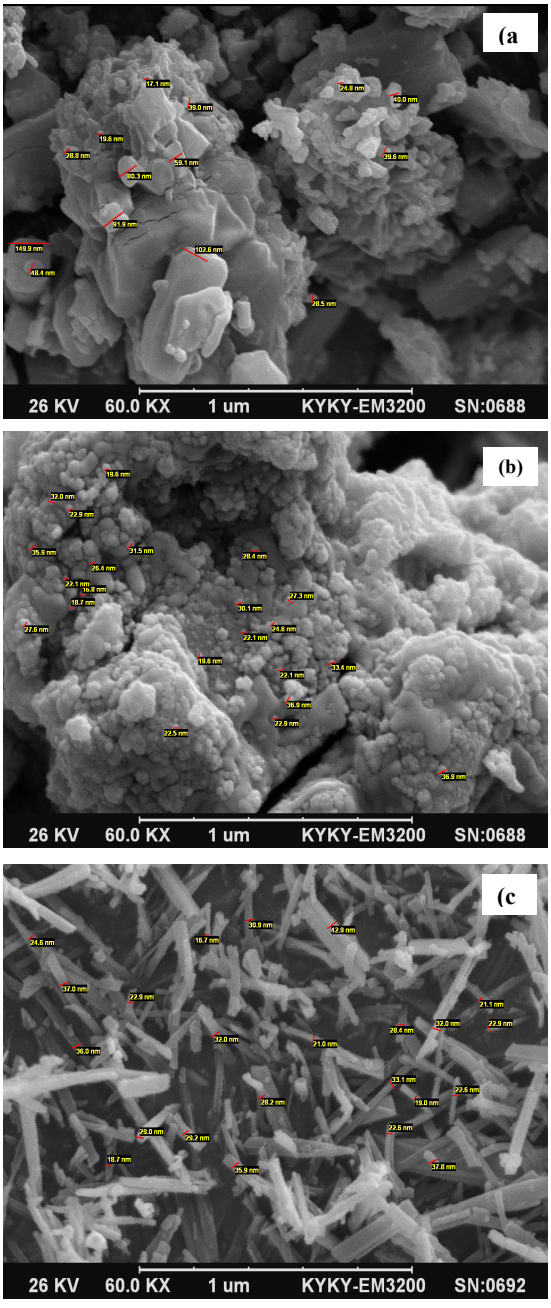

Fig. 2. SEM images of the (a) as-prepared (b) annealed FeCe nanoparticles at 500°C and (c) annealed one at 1100°C.

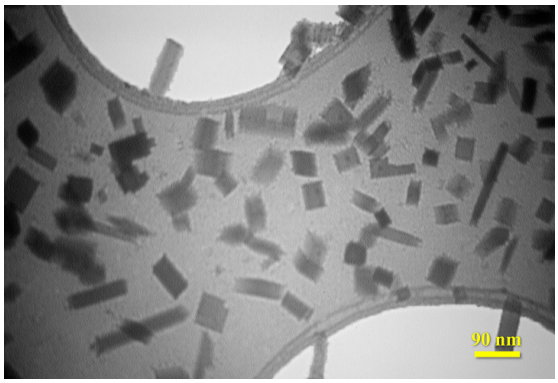

Fig. 3. TEM image of the as-prepared FeCe nanoparticles

SEM analysis was used for the morphological study of nanoparticles of samples. It can be seen that, with increasing temperature size of the particles decreased from 45 nm to 32 nm. Fig. 2(a) shows the SEM image of the as-prepared FeCe nanoparticles prepared by this method. In this figure, the particles prepared with formation of clusters. Fig. 2(b) shows the SEM image of the annealed FeCe nanoparticles at 500°C for 3 hours. Fig. 2(c) shows the SEM image of the annealed sample at 1100°C for 3 hours. As it can be seen the nano-rods particles were formed with less aggregation (Sebt, *et al.*, 2009). The particle size of as-prepared samples was measured about 45 nm and crystallite size of annealed nanocrystals about 32 nm and 30 nm for samples at 500°C and 1100°C in diameter respectively.

The transmission electron microscopic (TEM) analysis was carried out to confirm the actual size of the particles, their growth pattern and the distribution of

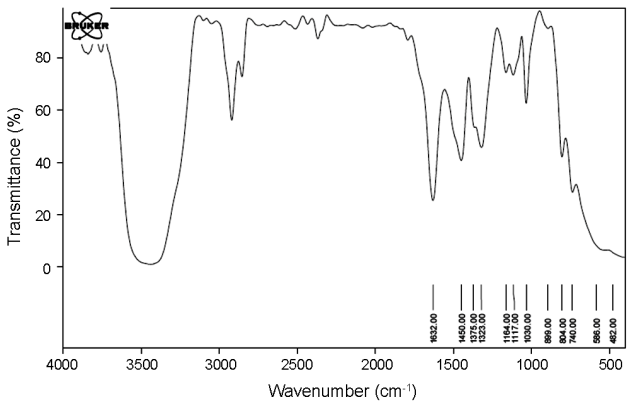

Fig. 4. FTIR spectrum of as-prepared FeCe sample

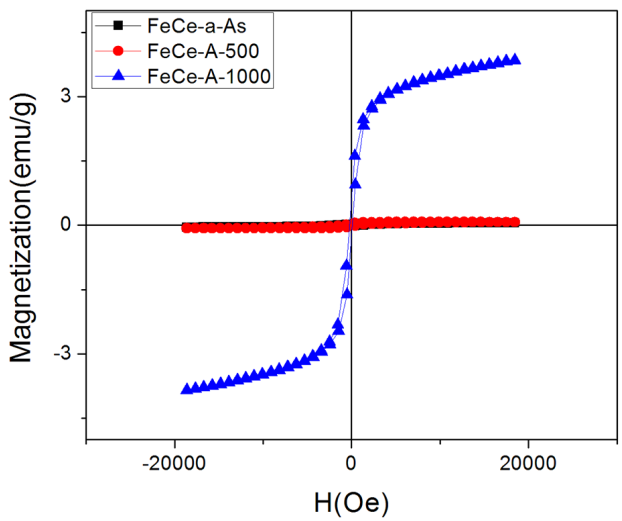

Fig. 5. VSM loops at 300 K for representative Fe-Ce samples in different temperature

the crystallites. Fig. 3 shows the as-synthesized TEM image of squared-like FeCe nanoparticles with average diameter of 45 nm prepared by chemical reduction route.

In Fig. 4, the infrared spectrum (FTIR) of the synthesized FeCe nanoparticles was in the range of 400-4000 cm^{-1} wavenumber which identify the chemical bonds as well as functional groups in the compound. The large broad band at 3450 cm^{-1} is ascribed to the O-H stretching vibration in OH^- groups. The absorption peaks around 1632 cm^{-1}, 1450 cm^{-1} are due to the asymmetric and symmetric bending vibration of C=O. The strong band below 700 cm^{-1} is assigned Fe-Ce stretching mode. The bands corresponding to Fe-Ce stretching mode are seen at 586 cm^{-1} and 482 cm^{-1}.

Magnetizations M versus applied magnetic field H for powders of the samples are measured at room temperature by cycling the magnetic field between -20k to 20k G. The magnetization curve in Fig. 5 shows hysteresis behavior for as-synthesized samples and annealed one at 500°C and 1100°C. Magnetic measurements show a coercive field around 1632 G for as-prepared and saturation magnetism of 3.79 emu/g for annealed one at 1100°C.

CONCLUSIONS

Fe_2O_3-CeO_2 nanocomposites have been successfully synthesized using iron nitrate and cerium nitrate in the presence of CTAB surfactant. XRD spectrum showed cubic structure of the samples. SEM images indicated that with increasing temperature the size of the nanoparticles decreased with less agglomeration. TEM image exhibited that the as-synthesized FeCe nanoparticles with an average diameter about 45 nm with good uniformity. FTIR spectra detected the presence of Fe-Ce stretching mode in the samples. Magnetic measurements studies showed a good coercive field and saturation magnetism around 1632 G and 3.79 emu/g.

REFERENCES

FARAHMANDJOU, M., (2016). Magnetocrystalline properties of Iron-Platinum (L1o-FePt) nanoparticles through phase transition. Iran. J. Phys. Res., 16: 1-5.

FARAHMANDJOU, M. & KHALILI, P., (2013). Morphology Study of anatase nano-TiO_2 for Self-cleaning Coating. Int. J. Fund. Phys. Sci., 3: 54-56.

FARAHMANDJOU, M. & RAMAZANI, M., (2015). Fabrication and Characterization of Rutile TiO_2 Nanocrystals by Water Soluble Precursor. Phys. Chem. Res., 3: 293-298.

JURABLU, S., FARAHMANDJOU, M. & FIROOZABADI, T.P., (2015). Multiple-layered structure of obelisk-shaped crystalline nano-ZnO prepared by sol–gel route. J. Theoretical. Appl. Phys., 9: 261–266.

FARAHMANDJOU, M. & SOFLAEE, F., (2015). Polymer-Mediated Synthesis of Iron Oxide (Fe_2O_3) Nanorods. Chin. J. Phys., 53: 080801.

FARAHMANDJOU, M. & SOFLAEE, F., (2015). Synthesis and characterization of α-Fe_2O_3 nanoparticles by simple co-precipitation method. Phys. Chem. Res., 3: 193-198.

SHADROKH, S., FARAHMANDJOU, M. & FIROOZABADI, T.P., (2016). Fabrication and Characterization of Nanoporous Co Oxide (Co3O4) Prepared by Simple Sol-gel Synthesis. Phys. Chem. Res., 4: 153-160.

KHODADADI, A., FARAHMANDJOU, M. & YAGHOUBI, M., (2019). Investigation on synthesis

and characterization of Fe-doped Al2O3 nanocrystals by new sol–gel precursors, Mater. Res. Express., 6: 025029.

FARAHMANDJOU, M., (2012). Synthesis of ITO Nanoparticles Prepared by Degradation of Sulfide Method. Chin. Phys. Lett., 29: 077306.

FARAHMANDJOU, M. & GOLABIYAN, N., (2016). Synthesis and characterization of Alumina (Al2O3) nanoparticles prepared by simple sol-gel method. Int. J. Bio-Inorg. Hybr. Nanomater., 5: 73-77.

FARAHMANDJOU, M., (2010). Synthesis and Structural Study of L10- FePt nanoparticles. Turk. J. Engin. Environ. Sci., 34: 265-270.

AKHTARI, F., ZORRIASATEIN, S., FARAHMANDJOU, M. & ELAHI, S.M., (2018). Structural, optical, thermoelectrical, and magnetic study of Zn1-xCoxO ($0 \leq x \leq 0.10$) nanocrystals. Int. J. Appl. Ceram. Technol., 15: 723-733.

AKHTARI, F., ZORRIASATEIN, S., FARAHMANDJOU, M. & ELAHI, S.M., (2018). Synthesis and optical properties of Co2+-doped ZnO Network prepared by new precursors. Mater. Res. Express., 5: 065015.

KHOSHNEVISAN, B., MARAMI, M. & FARAHMANDJOU, M., (2018). Fe3+-Doped Anatase TiO_2 Study Prepared by New Sol-Gel Precursors. Chin. Phys. Lett., 35: 027501.

MARAMI, M., FARAHMANDJOU, M. & KHOSHNEVISAN, B., (2018). Solgel Synthesis of Fe-doped TiO_2 Nanocrystals, J. electron. Mater.., 47 (2018) 3741-3749.

FARAHMANDJOU, M., HONARBAKHSH, S. & BEHROUZINIA, S., (2018). FeCo Nanorods Preparation Using New Chemical Synthesis. J. Supercond. Nov. Magn., 31: 4147–4152.

FARAHMANDJOU, M. & KHLILI, P., (2013). Study of Nano SiO_2/TiO_2 Superhydrophobic Self-Cleaning Surface Produced by Sol-Gel. Aust. J. Basic. Appl. Sci., 7: 462-465.

JURABLU, S., FARAHMANDJOU, M. & FIROOZABADI, T.P., (2015). Sol-gel synthesis of zinc oxide (ZnO) nanoparticles: study of structural and optical properties. J. Sci. Islamic Republic of Iran., 26: 281-285.

FARAHMANDJOU, M., (2013). The study of electro-optical properties of nanocomposite ITO thin films prepared by e-beam evaporation. Revista mexicana de física., 59: 205-207.

FARAHMANDJOU, M., HONARBAKHSH, S. & BEHROUZINIA, S., (2016). PVP-Assisted Synthesis of Cobalt Ferrite (CoFe2O4) Nanorods. Phys. Chem. Res., 4: 655-662.

JAFARI, A., KHADEMI, S. & FARAHMANDJOU, M., (2018). Nano-crystalline Ce-doped TiO_2 Powders: Sol-gel Synthesis and Optoelectronic Properties. Mater. Res. Express., 5: 095008.

LIU, W. & FLYTZANI, M., (1996). Transition metal-promoted oxidation catalysis by fluorite oxides: a study of CO oxidation over Cu-CeO_2. Chem. Engin. J. Biochem. Engin. J., 64: 283–294.

ZARINKAMAR, M.; FARAHMANDJOU, M. & FIROOZABADI, T.P., (2016). Diethylene Glycol-Mediated Synthesis of Nano-Sized Ceria (CeO_2) Catalyst. J. Nanostruct., 6: 114-118.

ZARINKAMAR, M.; FARAHMANDJOU, M. & FIROOZABADI, T.P., (2016). One-step synthesis of ceria (CeO_2) nano-spheres by a simple wet chemical method. J. Ceram. Proc. Res., 17: 166-169.

Gr ATZEL, M., (2001). Photoelectrochemical cells," Nature, 414: 338–344.

AVILA, H.J., JAIN, P., SEN, S. & KIM, S., (2010). Oxygen transport in Sc-doped CeO_2: cation (45Sc) NMR as a probe of anionic conductivity. Chem. Mater., 22: 893–897.

FARAHMANDJOU, M. & DASTPAK, M., (2018). Fe-Loaded CeO_2 Nanosized Prepared by Simple Co-Precipitation Route. Phys. Chem. Res., 6: 713-720.

DASTPAK, M., FARAHMANDJOU, M. & FIROOZABADI, T.P., (2016). Synthesis and preparation of magnetic Fe-doped CeO_2 nanoparticles prepared by simple sol-gel method. J. Supercond Nov. Magn., 29: 2925-2929.

ARA, J., MIRANDA, M., DOA, J. M., HERRERA, J.A. & PEA, J., (2001). Photocatalytic degradation of formic acid using Fe/TiO_2 catalysts: the role of Fe3+/Fe2+ ions in the degradation mechanism. Appl. Catal., B., 32: 49–61.

YAN, Q.Z., S.U, X.T., HUANG, Z.Y., (2006). Sol-gel autoigniting synthesis and structural property of

cerium-doped titanium dioxide nanosized powders. J. Europ. Ceram. Soc., 26: 915–921.

TERRIBILE, D., TROARELLI, A., LlORCA, J., De. C. & DOLCETTI, G., (1998). The synthesis and characterization of mesoporous high-surface area ceria prepared using a hybrid organic/inorganic route. J. Catal., 178: 299–308.

SCHERRER, P., (1918). Bestimmung der Grosse und der Inneren Struktur von Kolloidteilchen Mittels Rontgenstrahlen, Nachrichten von der Gesellschaft der Wissenschaften. Gottingen. Mathematisch-Physikalische Klasse. 2:, 98-100.

SEBT, S.A., PARHIZGAR, S.S., FARAHMANDJOU, M., ABEROMAND, P. & AKAVAN, M., (2009). HYPERLINK "javascript:void(0)" The role of ligands in the synthesis of FePt nanoparticles. J. Supercond. Nov. Magn., 22: 849-854.

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