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Chemical precipitation and characterization of multicomponent Perovskite Oxide nanoparticles – possible cathode materials for low temperature solid Oxide fuel cell

ABSTRACT

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A set of multicomponent perovskite oxide nanoparticles based on $La_{1-x}Sr_{x}Co_{1-y}Fe_{y}O_{3-\delta}(LSCF)$ were prepared by a simple chemical precipitation method for application in low temperature solid oxide fuel cells (LT-SOFC) as cathode materials. The precursor materials used in this synthesis were lanthanum nitrate hexahydrate [La(NO₃)₃.6H₂O], strontium nitrate $[Sr(NO_3)_2],$ cobalt nitrate hexahydrate [Co(NO₃)₂.6H₂O], ferric nitrate nonahydrate [Fe(NO₃)₃.9H₂O] [as basic materials] and sodium hydroxide [as precipitator material] and PVA [as surfactant]. Throughout the experiment, pH was maintained as pH > 9 by the addition of alkali (NaOH). A mixture of metal hydroxides (brown coloured) was formed when the aqueous mixture of basic materials mixed with the aqueous precipitant (NaOH) solution in proper stoichiometric compositions. The resultant hydroxide mixture was washed with ethanol and water mixture (1:9 volume %) to remove any unwanted impurities present along with the precipitate. The purified precipitate was dried at 50 - 100°C and heat treated at 300°C, 450°C, 600°C and 750°C for 2 hours each to get phase pure composite oxide powder. The resultant powder was characterized with TGA / DTA, XRD, FT-IR, Particle Size analysis and SEM. From the results, it was found that the chemical precipitation can be used effectively to prepare phase pure multicomponent perovskite oxides for application in SOFCs.

Keywords: Chemical precipitation, Multicomponent Perovskite Oxide nanoparticles, Cathode materials, SOFC.

INTRODUCTION

One of the driving forces behind the research effort directed towards SOFC technology is a lowering of the operating temperature of the fuel cell from 1000°C to around 600°C (low temperature range). However, to enable this, new materials with good lower-temperature performance in terms of level of conductivity, chemical and mechanical compatibility have to be identified and developed.

Manganite based perovskite oxides such as $La_{1-x}Sr_{x}MnO_{3-\delta}$ (LSM) have been widely used as cathodes due to their stability at high temperatures and thermal expansion compatibility with yttria stabilized zirconia(YSZ). These manganite based perovskites have relatively good stability at high temperature; however during long annealing times, pyrochlores $(La_2Zr_2O_7)$ are reported to form at the boundary between LSM and YSZ[1-2]. Better electrode materials that are less reactive with adjoining electrolyte are required. Moreover, to reduce the operating temperature of the SOFCs, a search for new cathode materials is important.La₁₋ $_{x}Sr_{x}Co_{1-y}Fe_{y}O_{3}$ (LSCF) is attracting substantial interest as a promising cathode material for LT-SOFCs because of its excellent properties, such as chemical and thermal stabilities, high catalytic activity for the oxygen reduction and high electrical conductivity [3].LSCF materials may replace the conventional LSM materials [4-7].LSCF based materials were synthesized by combustion synthesis [8-9], complexing method [10], sol-gel process [11], etc. In the present investigation, we report a simple wav synthesizing multicomponent nanoparticles perovskite oxide such as. $La_{0.9}Sr_{0.1}Co_{0.9}Fe_{0.1}O_{3-\delta}$ $La_{0.8}Sr_{0.2}Co_{0.9}Fe_{0.1}O_{3-\delta}$ La_{0.9}Sr_{0.1}Co_{0.8}Fe_{0.2}O_{3-δ} La_{0.8}Sr_{0.2}Co_{0.8}Fe_{0.2}O_{3-δ}by low temperature chemical precipitation method for possible application as cathode materials in LT-SOFC. The prepared nanoparticles are systematically characterized by TGA, XRD, FTIR, Particle Size analysis and SEM techniques.

EXPERIMENTAL

Materials and methods

Lanthanum nitrate hexahydrate $(La(NO_3)_3.6H_2O, 99\%, Loba Chemie)$, Strontium nitrate $(Sr(NO_3)_2, 99\%, Nice)$, cobalt nitrate non hydrate $(Co(NO_3)_2.9H_2O, \ge 97\%, Merck)$,iron

nitrate nonahydrate (Fe(NO₃)₃·9H₂O, 98%, Fisher Scientific), Sodium hydroxide (NaOH, \geq 97%, Merck) and Poly vinyl alcohol (PVA, 99% Merck)were used as starting materials. Deionized water was used for all the experiments.

Preparation of $La_{1-x}Sr_xCo_{1-y}Fe_yO_{3-\delta}$ (LSCF) nano ceramic particles by chemical precipitation method

In the typical experiment, the aqueous solutions of lanthanum nitrate hexahydrate, strontium nitrate, cobalt nitrate hexahydrate, ferric nitrate nonahydrate [as basic materials] and sodium hydroxide [as precipitator material] were prepared in distilled water. The later solution was mixed 2 ml of 10 % poly vinyl alcohol (as surfactant material). Initially, the aqueous metal nitrate solutions were added slowly drop-wise to the sodium hydroxide solution. They were mixed perfectly by a magnetic stirring apparatus (1000rpm) at room temperature for an hour. Throughout the experiment, the pH was maintained as pH>9 by the addition of alkali. The resultant brown coloured precipitate $[La(OH)_3+Sr(OH)_2+Co(OH)_2+Fe(OH)_3 \text{ with } PVA]$ was filtered and then washed with deionized water and ethanol (9:1) for 2 to 3 times and dried at 50 -100 °C for 2 to 3 hours. The resultant material was heat treated at 300°C, 450°C, 600°C and 750°C for 2 hours each to get a phase pure product. The amount of precursor materials used for the preparation of La_{1-x}Sr_xCo_{1-y}Fe_yO_{3-δ}nanoparticles is indicated in Table 1. Figure 1 shows the schematic illustration of the synthesis of La_{1-x}Sr_xCo_{1-y}Fe_yO₃₋ δ nano particles by the chemical precipitation method. Main reactions involved in the preparation of $La_{0.9}Sr_{0.1}Co_{0.9}Fe_{0.1}O_{3-\delta}$ $La_{0.8}Sr_{0.2}Co_{0.9}Fe_{0.1}O_{3-\delta}$ $La_{0.9}Sr_{0.1}Co_{0.8}Fe_{0.2}O_{3-\delta}$ and $La_{0.8}Sr_{0.2}Co_{0.8}Fe_{0.2}O_{3-\delta}$ δ occur during the experimental procedure can be written briefly as follows:

Reaction mechanism involved in the preparation of $La_{0.9}Sr_{0.1}Co_{0.9}Fe_{0.1}O_{3-\delta}$:

 $\begin{array}{c} 5.0 \text{ NaOH}_{(s)} \rightarrow 5.0 \text{ Na}^+_{(aq)} + 5.0 \text{ OH}^-_{(aq),} \\ 0.9 \text{ La}(\text{NO}_3)_3.6\text{H}_2\text{O}_{(s)} \rightarrow 0.9\text{La}^{3+}_{(aq)} + 2.7 \text{ NO}_3^-_{(aq)} + 6\text{H}_2\text{O}_{(aq),} \\ 0.1 \text{ Sr}(\text{NO}_3)_2 \rightarrow 0.1\text{Sr}^{3+}_{(aq)} + 0.2 \text{ NO}_3^-_{(aq),} \\ 0.9 \text{ Co}(\text{NO}_3)_2.6\text{H}_2\text{O}_{(s)} \rightarrow 0.9\text{Co}^{3+}_{(aq)} + 1.8 \text{ NO}_3^-_{(aq)} + 6\text{H}_2\text{O}_{(aq),} \\ 0.1 \text{ Fe}(\text{NO}_3)_3.9\text{H}_2\text{O}_{(s)} \rightarrow 0.1\text{Fe}^{3+}_{(aq)} + 0.3 \text{ NO}_3^-_{(aq)} + 9\text{H}_2\text{O}_{(aq),} \\ 0.9 \text{ La}^{3+}_{(aq)} + 0.1\text{Sr}^{3+}_{(aq)} + 0.9\text{Co}^{3+}_{(aq)} + 0.1\text{Fe}^{3+}_{(aq)} + 5.0\text{OH}^-_{(aq)} \rightarrow \end{array}$

 $\begin{array}{c} 0.9 \text{ La}(\text{OH})_{3.x} \text{ H}_2\text{O}_{(s)} \downarrow + 0.1 \text{ Sr}(\text{OH})_{2.x} \text{H}_2\text{O}_{(s)} \downarrow + 0.9 \text{ Co}(\text{OH})_{2.x} \text{ H}_2\text{O}_{(s)} \downarrow + 0.1 \text{ Fe}(\text{OH})_{3.x} \text{H}_2\text{O}_{(s)} \downarrow \\ \downarrow 50\text{-}100^{\circ} \text{ C} \\ 0.9 \text{ La}(\text{OH})_{3(s)} + 0.1 \text{ Sr}(\text{OH})_{2(s)} + 0.9 \text{ Co}(\text{OH})_{2(s)} + 0.1 \text{ Fe}(\text{OH})_{3(s)} + x \text{ H}_2\text{O}_{(g)} \uparrow \\ \downarrow 300^{\circ} \text{ C} / 450^{\circ} \text{ C} / 600^{\circ} \text{ C} / 750^{\circ} \text{ C} \text{ for } 2h \\ 0.9 \text{ La}(\text{OH})_{3(s)} + 0.1 \text{ Sr}(\text{OH})_{2(s)} + 0.9 \text{ Co}(\text{OH})_{2(s)} + 0.1 \text{ Fe}(\text{OH})_{3(s)} \rightarrow \text{La}_{0.9}\text{Sr}_{0.1}\text{Co}_{0.9}\text{Fe}_{0.1}\text{O}_{3.\delta} \end{array}$

Reaction mechanism involved in the preparation of $La_{0.8}Sr_{0.2}Co_{0.9}Fe_{0.1}O_{3-\delta}$:

Reaction mechanism involved in the preparation of $La_{0.9}Sr_{0.1}Co_{0.8}Fe_{0.2}O_{3-\delta}$:

Reaction mechanism involved in the preparation of $La_{0.8}Sr_{0.2}Co_{0.8}Fe_{0.2}O_{3-\delta}$:

Sample	Molarity of	Molarity of	Molarity of	Molarity of	Molarity of
	(La(NO ₃) ₃ /	Sr(NO ₃) ₂ /	Co(NO ₃) ₂ /	Fe(NO ₃) ₃ /	NaOH /
	Amount (g)	Amount (g)	Amount (g)	Amount (g)	Amount (g)
$La_{0.9}Sr_{0.1}Co_{0.9}Fe_{0.1}O_{3-\delta}$	0.09 M /	0.01 M /	0.09 M /	0.01 M /	0.5 M /
	3.897 g	0.211 g	2.619 g	0.404 g	2.0 g
$La_{0.8}Sr_{0.2}Co_{0.9}Fe_{0.1}O_{3\text{-}\delta}$	0.08 M /	0.02 M /	0.09 M /	0.01 M/	0.49 M /
	3.46 g	0.423 g	2.619 g	0.404 g	1.96 g
$La_{0.9}Sr_{0.1}Co_{0.8}Fe_{0.2}O_{3-\delta}$	0.09 M /	0.01 g /	0.02 M /	0.02 M /	0.51 M /
	3.897 g	0.211 g	0.808 g	0.808 g	2.04 g
$La_{0.8}Sr_{0.2}Co_{0.8}Fe_{0.2}O_{3-\delta}$	0.08 M /	0.02 M /	0.02 M /	0.02 M /	0.51 M /
	3.46 g	0.423 g	0.808 g	0.808 g	2.04 g

Table 1. Amount of precursor materials (dissolved in 100 ml of water each) used for the preparation of $La_{1-x}Sr_xCo_{1-y}Fe_yO_{3-\delta}$ nanoparticles by chemical precipitation method



Fig. 1. Schematic representation of the synthesis of $La_{1-x}Sr_xCo_{1-y}Fe_yO_{3-\delta}$ nanoparticles by chemical precipitation method

Characterization of the samples

Thermal analysis the of precursor precipitate was performed with Perkin Elmer TGA 7 instrument under nitrogen atmosphere at a heating rate of 10° C /minute. The powder XRD studies were carried out using a Shimadzu XRD6000 X-ray diffractometer at a scan speed of 5 degrees minute⁻¹ using CuKa radiation. The theoretical density of the powders was calculated with the obtained XRD data. The crystallite sizes of the powder were calculated by Scherrer's formula. Bruker IFS 66V FT-IR spectrometer was employed to record the FTIR spectra of doped CeO₂ powders in the range of 4000 - 400 cm⁻¹. The crystallite sizes of the ceramic powders were calculated by Scherrer's formula. The particle size of the powder was measured using Malvern Particle Size Analyzer using triple distilled water as medium. The surface morphology of the particles was studied by means of JEOL Model JSM-6360 scanning electron microscope.

RESULTS AND DISCUSSION

TGA studies of precursor materials

precipitate The dried precursor [La(OH)₃+Sr(OH)₂+Co(OH)₂+Fe(OH)₃withPVA] with an initial mass 5-9 mg was placed in an open platinum crucible. The mass scale of the instrument was calibrated with standard reference materials based on the measurement of curie Points (Tc) of alumel alloy (Tc = 427.35 K) and nickel (Tc = 628.45 K). The TGA patterns obtained with the precipitate precursor materials $[La(OH)_3+Sr(OH)_2+Co(OH)_2+Fe(OH)_3with PVA]$ are indicated in Figure 2 (a- d). From Figure 2 (ad), it was understood the total weight loss was found to be in the range of 24 - 31% from the temperature of 25 to 700° C. The initial weight loss of about 2 - 3 % observed at around 100 °C in all the samples may be due to the removal of water molecule. The total weight loss of about 10 - 13 % observed at 300°Cin all the samples may be attributed to the removal of oxides of carbon. Further weight loss found in the samples until 700° C may due to the decomposition of remaining organics and other impurities. From the TGA curves of all the samples, it was understood that the weight of the sample reduced gradually from 25 -700° C. Hence, the TGA results inferred that the

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resultant precipitate (mixture of hydroxides and surfactant) should be heat treated above 700° C gradually in order to remove the impurities from the sample and to get phase pure material.





The powder XRD patterns obtained on $La_{0.9}Sr_{0.1}Co_{0.9}Fe_{0.1}O_{3-\delta}$ $La_{0.8}Sr_{0.2}Co_{0.9}Fe_{0.1}O_{3-\delta}$ $La_{0.9}Sr_{0.1}Co_{0.8}Fe_{0.2}O_{3-\delta}$ and $La_{0.8}Sr_{0.2}Co_{0.8}Fe_{0.2}O_{3-\delta}$ nanoparticles prepared by chemical precipitation method are indicated in Figure 3 (a-d) respectively. The XRD peaks are found to be very sharp indicating the highly crystalline nature of the perovskite. Since the standard XRD data for La₁- $_{x}Sr_{x}Co_{1-y}Fe_{y}O_{3-\delta}$ is not available in the literature, the obtained XRD data were compared with the XRD data of standard JCPDS pattern for LaCoO₃ (JCPDS card No. 25-1060). The obtained'd' values for LSCF were in match with the reported data for LaCoO₃. The XRD patterns of the heat-treated powders reveal the formation of well-crystalline single phase perovskite structure with rhombohedral (hexagonal) cell geometry. The lattice parameters are calculated from 2θ values in the X-ray diffraction patterns and the data is indicated in Table 2. The theoretical density (D_X) for the samples was calculated according to the formula [12]:

$$D_X = \frac{Z \times M}{N \times a^3}$$
 g. cm⁻³

Where, Z = number of chemical species in the unit cell, M = molecular mass of the sample (g/mol), N = Avogadro's number (6.022×10^{23}) and a = lattice constant (cm). Crystallite size of the samples was calculated from XRD line broadening method using the following Scherrer relationship [13]:

$$\mathbf{D}_{\mathrm{p}} = \frac{k\lambda}{\beta cos\theta}$$

Where 'D_p' is the crystallite size, 'k' is a numerical constant (~0.9), 'λ' is the wavelength of X-rays (for CuKα radiation, $\lambda = 1.5418$ Å), 'β' is the effective broadening taken as a full width at half maximum (FWHM) (in radians), 'θ' is the diffraction angle for the peak. The crystallographic parameters obtained on La_{1-x}Sr_xCo_{1-y}Fe_yO_{3-δ} nanoparticles are given in Table 2.The crystallite size values were found in the range of 8.29 – 10.79 nm. The theoretical density values were found to be in the range of 6.8 – 7.1g/cc.



Fig. 3. Powder XRD patterns obtained on (a) $La_{0.9}Sr_{0.1}Co_{0.9}Fe_{0.1}O_{3.\delta}(b) La_{0.8}Sr_{0.2}Co_{0.9}Fe_{0.1}O_{3.\delta}(c) La_{0.9}Sr_{0.1}Co_{0.8}Fe_{0.2}O_{3.\delta}(d) La_{0.8}Sr_{0.2}Co_{0.8}Fe_{0.2}O_{3.\delta}(d) La_{0.8}Sr_{0.2}Co_{0.8}Fe_{0.2}O_{0.8}Fe_{0.2}O_{0.8}Fe_{0.2}O_{0.8}Fe_{0.2}O_{0.8}Fe_{0.2}O_{0.8}Fe_{0.2}O$

Sample	Crystal structure	Unit cell lattice parameters (Å)	Unit cell volume (Å) ³	Theoretical density (g/cc)	Crystallite Size (nm)
Standard XRD data for LaCoO ₃ powder (JCPDS No. 25-1060)	Rhombohedral (Hexagonal)	a = 5.441 c =13.088	335.54	7.299	
$La_{0.9}Sr_{0.1}Co_{0.9}Fe_{0.1}O_{3\text{-}\delta}$	Rhombohedral (Hexagonal)	a = 5.4340 c = 13.119	335.49	7.114	8.29
$La_{0.8}Sr_{0.2}Co_{0.9}Fe_{0.1}O_{3\text{-}\delta}$	Rhombohedral (Hexagonal)	a = 5.4445 c= 13.259	340.73	6.855	9.46
$La_{0.9}Sr_{0.1}Co_{0.8}Fe_{0.2}O_{3\text{-}\delta}$	Rhombohedral (Hexagonal)	a = 5.4487 c = 13.101	336.83	7.077	9.81
$La_{0.8}Sr_{0.2}Co_{0.8}Fe_{0.2}O_{3\text{-}\delta}$	Rhombohedral (Hexagonal)	a = 5.4498 c = 13.087	336.61	6.929	10.79

Table 2. The crystallographic parameters obtained on $La_{1-x}Sr_xCo_{1-y}Fe_yO_{3-\delta}$ nanoparticles

FTIR studies

Figure 4 (a- d) show the FTIR spectra obtained on $La_{0.9}Sr_{0.1}Co_{0.9}Fe_{0.1}O_{3-\delta}$, $La_{0.8}Sr_{0.2}Co_{0.9}Fe_{0.1}O_{3-\delta}$, $La_{0.9}Sr_{0.1}Co_{0.8}Fe_{0.2}O_{3-\delta}$ and $La_{0.8}Sr_{0.2}Co_{0.8}Fe_{0.2}O_{3-\delta}$ nanoparticles prepared by chemical precipitation method. The broader peak appeared at ~600 cm⁻¹ is characteristic of the MO₆octahedra commonly found in perovskite

oxide powder and is observed in this system too [14]. The bands appeared at ~ 600 cm⁻¹ and ~650 cm⁻¹ in LSCF samples are ascribed to Co-O / Fe-O and BO₆ stretch vibrations in the perovskite structure, respectively [15]. The peaks found near to 1600 cm⁻¹ in the samples are due to the presence of atmospheric moisture as reported earlier[16].



Fig. 4. FTIR spectra obtained on (a) $La_{0.9}Sr_{0.1}Co_{0.9}Fe_{0.1}O_{3.\delta}$ (b) $La_{0.8}Sr_{0.2}Co_{0.9}Fe_{0.1}O_{3.\delta}$ (c) $La_{0.9}Sr_{0.1}Co_{0.8}Fe_{0.2}O_{3-\delta}$ (d) $La_{0.8}Sr_{0.2}Co_{0.8}Fe_{0.2}O_{3-\delta}$ nanoparticles prepared by chemical precipitation method

Particle characteristics

The prepared La_{1-x}Sr_xCo_{1-v}Fe_vO₃₋ _δnanoparticles (after calcination at 750° C for 2 h) were subjected to particle size measurements using Malvern particle size analyzer with triple distilled water as medium. For all the measurements, 0.30 g of sample was sonicated in 30 ml triple distilled water for about 10 minutes and after that the sample was subjected for particle size analysis. The particle size distribution curves of $La_{0.9}Sr_{0.1}Co_{0.9}Fe_{0.1}O_{3-\delta}$, La_{0.8}Sr_{0.2}Co_{0.9}Fe_{0.1}O_{3-δ}, $La_{0.9}Sr_{0.1}Co_{0.8}Fe_{0.2}O_{3-\delta}$ and $La_{0.8}Sr_{0.2}Co_{0.8}Fe_{0.2}O_{3-\delta}$ nanoparticles prepared by chemical precipitation method are shown in Figure 5 (a- d). The particle characteristics data obtained on La_{1-x}Sr_xCo_{1-y}Fe_yO₃₋

 $_{\delta}$ nanoparticles particles is indicated in Table 3. The presence of bigger particles (> 500 nm) in the sample may be due to high temperature treatment.

SEM studies

The surface microstructure of La₁₋ $_xSr_xCo_{1-y}Fe_yO_{3-\delta}$ nanoparticles was studied with SEM. The SEM photographs of La_{1-x}Sr_xCo_{1-y}Fe_yO_{3- δ} nanoparticles are shown in Figure 6 (a- d). SEM analysis provides the information about the size and shape of the particle and pore. From the SEM studies, it was found that the grain size was in the range of 70 – 126 nm. Also, few larger particles are also present in the samples which are in accordance with the particle characteristics data.



 $\begin{array}{l} \label{eq:Fig. 5. Particle size curves obtained on (a) $La_{0.9}Sr_{0.1}Co_{0.9}Fe_{0.1}O_{3-\delta}$ (b) $La_{0.8}Sr_{0.2}Co_{0.9}Fe_{0.1}O_{3-\delta}$ (c) $La_{0.9}Sr_{0.1}Co_{0.8}Fe_{0.2}O_{3-\delta}$ (d) $La_{0.8}Sr_{0.2}Co_{0.8}Fe_{0.2}O_{3-\delta}$ nanoparticles prepared by chemical precipitation method \\ \end{array}$

Table 3. Particle characteristics data obtained on La_{1-x}Sr_xCo_{1-y}Fe_yO_{3-δ} nanoparticles prepared by chemical precipitation method

Sample	Peak 1		Peak 2		Average
	% Intensity	Diameter (nm)	% Intensity	Diameter (nm)	size (d.nm)
$La_{0.9}Sr_{0.1}Co_{0.9}Fe_{0.1}O_{3-\delta}$	100.0	626.3			1171
$La_{0.8}Sr_{0.2}Co_{0.9}Fe_{0.1}O_{3-\delta}$	100.0	469.6			387.5
$La_{0.9}Sr_{0.1}Co_{0.8}Fe_{0.2}O_{3-\delta}$	97.7	1037	2.3	5569	1327
$La_{0.8}Sr_{0.2}Co_{0.8}Fe_{0.2}O_{3\text{-}\delta}$	100.0	794.4			1176



Fig. 6. SEM photographs obtained on (a) $La_{0.9}Sr_{0.1}Co_{0.9}Fe_{0.1}O_{3.\delta}$ (b) $La_{0.8}Sr_{0.2}Co_{0.9}Fe_{0.1}O_{3.\delta}$ (c) $La_{0.9}Sr_{0.1}Co_{0.8}Fe_{0.2}O_{3.\delta}$ (d) $La_{0.8}Sr_{0.2}Co_{0.8}Fe_{0.2}O_{3.\delta}$ nanoparticles prepared by chemical precipitation method

CONCLUSIONS

Chemical precipitation method can be effectively used for the preparation of $La_{1-x}Sr_xCo_{1-y}Fe_yO_{3-\delta}$ based cathode materials for application in LT-SOFCs. TGA patterns obtained on precursor precipitate revealed the methodology to get phase pure $La_{1-x}Sr_xCo_{1-y}Fe_yO_{3-\delta}$ materials. The powder XRD data obtained is in agreement with the standard reported JCPDS data and crystallized as rhombohedral (hexagonal) cell geometry. From the FTIR spectra, it is observed that characteristic peak (~650 cm⁻¹) is present in all four samples. The presence of nano particles is confirmed by the particle size analysis and SEM measurements.

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