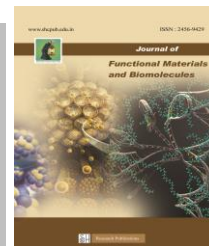




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Synthesis and Comparative Study of ZnO-CeO₂ and NiO-CeO₂ Nanocomposites

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Abstract

The ZnO-CeO₂ and NiO-CeO₂ nanocomposites are prepared by a simple chemical coprecipitation method. The prepared nanocomposites are confirmed by Powder XRD, EDAX, and FTIR. The powder XRD patterns of ZnO-CeO₂ and NiO-CeO₂ nanocomposites confirms the crystalline nature and the average crystallite size is found to be 13.9 nm and 8.9 nm for ZnO-CeO₂ and NiO-CeO₂ nanocomposites respectively. The HRSEM images show that the ZnO-CeO₂ nanocomposites have mixed hexagonal and spherical morphology. The rod and spherical shape morphology of NiO-CeO₂ nanocomposites is confirmed by HRSEM images. The EDAX analysis confirms the presence of Ce, Ni, Zn and O in the prepared nanocomposites. FTIR spectra of ZnO-CeO₂ and NiO-CeO₂ nanocomposites show the characteristics of metal oxide vibrations below 870 cm⁻¹.

Keywords: Nanocomposites, crystallite size, hexagonal, spherical shape.

1 Introduction

In recent years, worldwide there has been a lot of attentiveness in the research field, for studying nanocomposite materials due to enhanced mechanical stability, electrical and thermal conductivity [1-3]. Zinc oxide nanoparticle is a familiar semiconducting material with a wide energy gap. Nickel oxide nanoparticle is a natural p-type semiconductor with a wide energy gap and good thermal conductivity and chemical stability [4]. Cerium oxide (CeO₂) is a rare earth metal oxide, that has been focused on due to its excessive thermal strength, and oxygen storage capability [5]. ZnO-CeO₂ Nano-composites in a mixture part synthetic arrangement provide a systematic way to magnify the field emission performance of ZnO structures, such as chemical sensors, optoelectronic devices, and photocatalysts [5-8]. By fusing the properties of ZnO-CeO₂ the composite shows a good photocatalytic material. NiO-CeO₂ nanocomposite shows the properties such as high melting point, thermal conductivity, hardness and mechanical strength. The nanocomposites have been synthesized by various methods such as sol-gel, coprecipitation, solvothermal, hydrothermal, photochemical reduction, polymerization, combustion, laser ablation, and sonochemical method [9-12]. Compared with the above-

mentioned methods, the coprecipitation method is a simple, high purity, less expensive method [13-15]. The main objective of this research work is an investigation of the preparation and structural properties of ZnO-CeO₂ and NiO-CeO₂ nanocomposites by the coprecipitation method.

2 Experimental Sections

2.1 Preparation of ZnO-CeO₂ nanocomposites

All the reagents were of analytical grade and used without any further purification. 2.21 g of zinc acetate dihydrate (Zn(CH₃COO)₂ · 2H₂O), 4.34 g of cerium nitrate hexahydrate (Ce(NO₃)₃ · 6 H₂O) and 3.42 g of lactose (C₁₂H₂₂O₁₁) were added and dissolved in 100 ml double distilled. With the precursor solution, 0.2 M of sodium hydroxide (NaOH) was added drop-wise to the reaction mixture until pH=11. The obtained precipitate was collected and washed several times with distilled water and ethanol. Then the reaction mixture was heated at 80°C for 12 hours. Finally, it was calcinated under 500°C for 2 hours.

2.2 Preparation of NiO-CeO₂ nanocomposites

2.9 g of nickel nitrate hexahydrate (Ni(NO₃)₂ · 6 H₂O) and 4.34 g of cerium nitrate hexahydrate were added to 3.42 g of lactose (C₁₂H₂₂O₁₁) 100vml solution. After 30 minutes, sodium hydroxide (NaOH) was added drop-wise to the reaction mixture until pH=11. The obtained precipitate was collected and washed several times with distilled water and ethanol. Then the final product was heated at 80°C for 12 hours. Finally, it was calcinated under 500°C for 2 hours.

2.3 Characterizations

The crystalline nature of the prepared samples is studied by Rigaku powder X-ray diffractometer with CuKα (λ = 1.54187Å) radiation in the range of 10° – 80° at room temperature. The surface morphology of the prepared samples is analyzed by FEI Quanta FEG 200F high-resolution scanning electron microscope. The elemental confirmation study is carried out by an Energy Dispersive X-ray spectrometer. The FTIR spectra are recorded in the

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range of 400 - 4000 cm^{-1} by PERKIN ELMER SPECTRUM II FTIR spectrometer.

3. Results and Discussion

3.1 Powder X-ray Diffraction Analysis

Powder X-ray diffraction analysis is one of the useful methods, which is used to analyze the structure of crystalline materials for the prepared samples. The powder XRD pattern of ZnO-CeO₂ nanocomposite is shown in fig. 1(a). The XRD peaks are indexed using JCPDS # 65-6533 (ZnO) and JCPDS # 34 – 0394 (CeO₂). The average crystallite size is estimated using the Scherer equation. The crystallite size, strain and dislocation density of the synthesized ZnO-CeO₂ nanocomposite are depicted in Table 1. The average

crystallite size of the ZnO-CeO₂ nanocomposite is found to be 13.9 nm.

The powder XRD pattern of NiO-CeO₂ nanocomposite is shown in fig. 1(b). The XRD peaks are indexed using JCPDS # 89-5881 (NiO) and JCPDS # 34 – 0394 (CeO₂). The average crystallite size is estimated using the Scherer equation. The crystallite size, strain and dislocation density of the synthesized NiO-CeO₂ nanocomposite are presented in Table 2. The average crystallite size of the NiO-CeO₂ nanocomposite is found to be 8.9 nm. From the PXRD analysis, the NiO-CeO₂ nanocomposite has a large strain and dislocation density due to its smaller crystallite size.

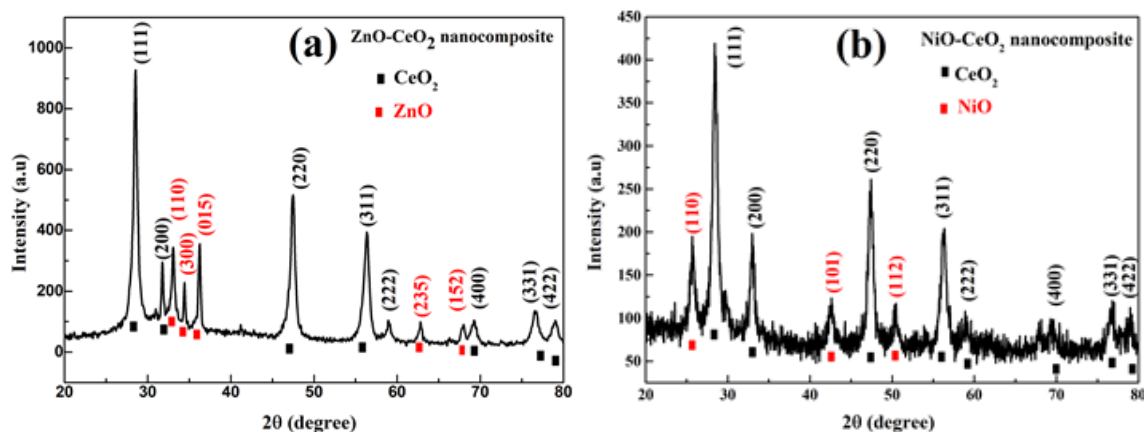


Fig. 1(a) Powder XRD pattern of ZnO-CeO₂ nanocomposites and 1(b) Powder XRD pattern for NiO-CeO₂ nanocomposites

Table 1 PXRD data of ZnO-CeO₂ nanocomposites

2θ (degree)	FWHM (radian)	Crystallite size (nm)	Strain	Dislocation density ($\times 10^{15} \text{ m}^{-2}$)
28.52	0.8345	10.27	0.0143	9.4811
31.84	1.0618	13.97	0.0162	5.1239
33.00	0.5738	15.09	0.0085	4.3915
34.37	0.7808	11.13	0.011	8.0725
36.33	0.6819	12.82	0.0091	6.0844
47.56	0.8464	10.72	0.0084	8.7018
56.31	0.8367	11.26	0.0068	7.8872
58.85	0.5904	16.15	0.0046	3.8340
69.28	0.5357	18.84	0.0034	2.8173
76.66	0.7366	14.37	0.0041	4.8426
78.91	0.5853	18.37	0.0031	2.9633

Table 2 PXRD data of NiO-CeO₂ nanocomposites

2θ (degree)	FWHM (radian)	Crystallite size (nm)	Strain	Dislocation density ($\times 10^{15} \text{ m}^{-2}$)
28.45	0.71085	12.05	0.0122	6.8869
32.99	0.56451	15.34	0.0083	4.2496
42.63	1.61627	5.51	0.0181	32.937
47.39	0.86248	10.51	0.0086	9.0530
50.16	1.30986	7.01	0.0122	20.408
56.28	0.8197	11.48	0.0067	7.5878
69.33	2.90952	3.47	0.0184	83.050
76.66	1.1872	8.91	0.0066	12.596
78.81	1.81332	5.92	0.0096	28.533

3.2 Morphological analysis

The different magnification of HRSEM images of ZnO-CeO₂ nanocomposites is shown in fig. 2. From the HRSEM images of ZnO-CeO₂ nanocomposite, the particles have sphere-like morphology with some agglomeration. The different magnification of HRSEM images of NiO-CeO₂ nanocomposite is shown in fig. 3. From the HRSEM images of NiO-CeO₂ nanocomposites, the particles have spherical morphology with large agglomeration. The particles of NiO-CeO₂ nanocomposite are a relatively smaller size than the ZnO-CeO₂ nanocomposite.

3.3 EDAX Analysis

The energy dispersive X-ray spectroscopic analysis is a useful technique that provides the elemental confirmation and quantitative compositional information of the prepared samples. Fig. 4 shows the EDAX spectra of (a) ZnO-CeO₂ nanocomposite and (b) NiO-CeO₂ nanocomposite. The EDAX spectrum of ZnO-CeO₂ nanocomposite confirms the presence of Zn, Ce and O. The EDAX spectrum of NiO-CeO₂ nanocomposite confirms the presence of Ni, Ce and O. The Quantitative information of the prepared ZnO-CeO₂ nanocomposite and NiO-CeO₂ nanocomposite are presented in Table 3

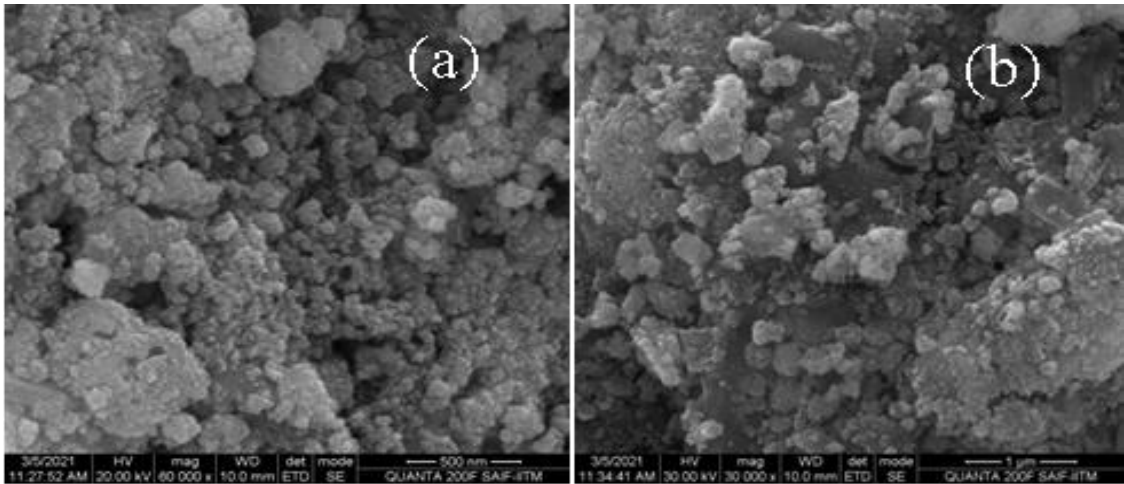


Fig 3. HRSEM images of NiO-CeO₂ nanocomposites at magnification (a) 60000x and (b) 30000x

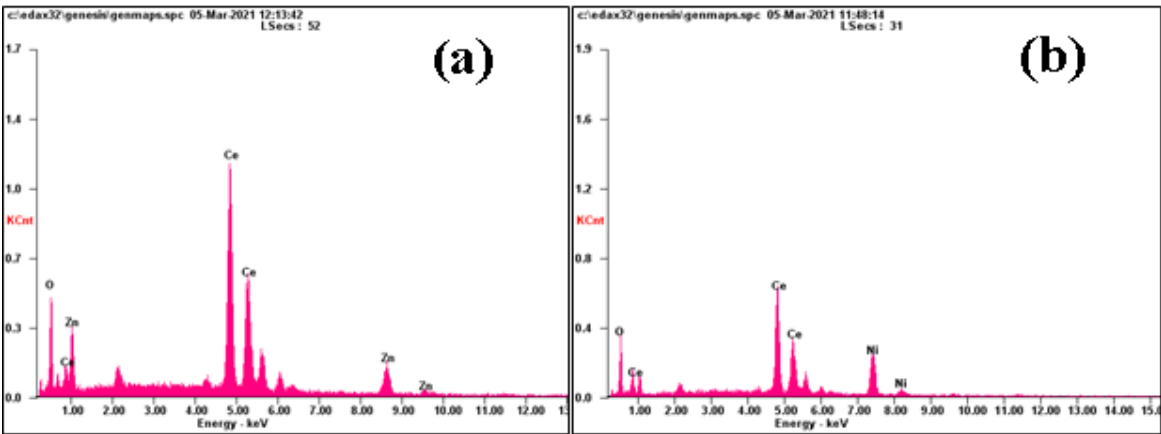


Fig 4. EDAX spectra of (a) ZnO-CeO₂ nanocomposite and NiO-CeO₂ nanocomposite

Table 3. EDAX Quantitative data of ZnO-CeO₂ nanocomposite and NiO-CeO₂ nanocomposite

Element	ZnO-CeO ₂ nanocomposite		NiO-CeO ₂ nanocomposite	
	Wt%	At%	Wt%	At%
OK	20.47	66.49	20.75	62.80
CeL	70.04	25.97	58.76	20.30
ZnK	09.48	07.54	-	-
NiK	-	-	20.49	16.90

3.4 FTIR Analysis

The FTIR Spectrum of ZnO-CeO₂ nanocomposite and NiO-CeO₂ nanocomposite is shown in fig. 5 and Fig. 6 respectively. From the FTIR spectra, the absorption peaks

around 3407 cm⁻¹, 1437 cm⁻¹, 1373 cm⁻¹ and 1059 cm⁻¹ are due to the OH stretching vibration of water molecules absorbed from the atmosphere[16-19]. The peak around 1568 cm⁻¹ is due to C-O vibrations. The peaks of around

862 cm^{-1} and 556 cm^{-1} are due to combined vibrations of Ce-O and Zn-O (fig. 5). The peak around 501 cm^{-1} is due to combined vibrations of Ce-O and Ni-O (fig. 6).

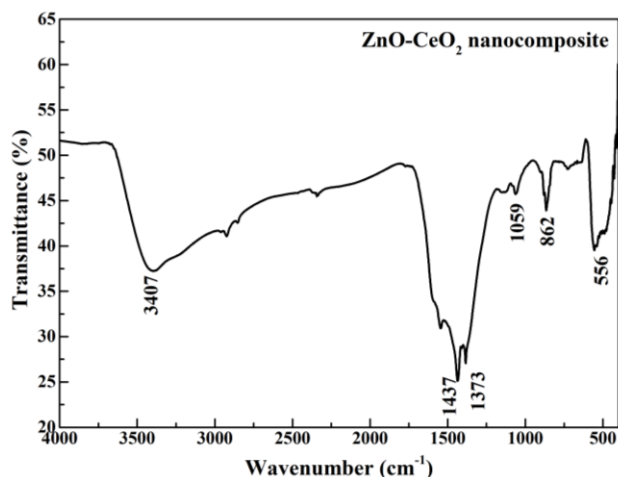


Fig 5. FTIR Spectrum of ZnO-CeO₂ nanocomposite

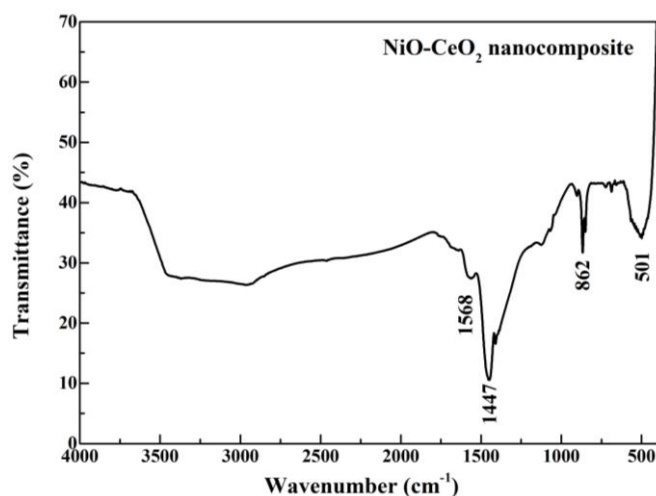


Fig. 6 (b) FTIR Spectrum of NiO-CeO₂ nanocomposite

4. Conclusion

ZnO-CeO₂ and NiO-CeO₂ nanocomposites have been successfully prepared by the co-precipitation method. The powder XRD study shows that the average crystallite size value is 13.9 nm and 8.9 nm for ZnO-CeO₂ nanocomposite and NiO-CeO₂ nanocomposite respectively. The EDAX study confirms the presence of Ce, Zn and O in ZnO-CeO₂ nanocomposite and Ce, Ni and O in NiO-CeO₂ nanocomposite. The FTIR spectra of ZnO-CeO₂ and NiO-CeO₂ nanocomposites show the characteristic metal oxide vibrations in the fingerprint region. The HRSEM analysis reveals that the ZnO-CeO₂ nanocomposite has sphere-like morphology and the NiO-CeO₂ nanocomposite has spherical morphology with agglomeration. The NiO-CeO₂ nanocomposite has smaller particles compared with the ZnO-CeO₂ nanocomposite. Hence, NiO-CeO₂ nanocomposites are the potential candidate for sensor and catalytic applications.

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