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Influence of Calcination Temperature on Physical and Optical Properties of Nickel Chromite Nanoparticles

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Abstract:

Nickel chromite nanoparticles were prepared by sol-gel auto combustion method followed by calcination at different temperatures, 700, 900, 1100 and 1300°C for 8h. The prepared nanoparticles were characterized by XRD, SEM, EDS, FTIR and UV-Vis spectroscopy. From the XRD patterns, the Miller indices (hkl) were identified for all the prominent peaks for all the samples, it was clear that the compound crystallized according to spinel structure with a space group of Fd3m. SEM images showed non-uniform agglomerated fragments having a lot of voids and pores. Using Scherrer method, the crystallite size was calculated and found gradually increasing trend with the calcination temperature from 13.73 to 22.33 nm, where as the average grain size was increased from 140.00 to 163.46 nm with calcination temperature. FTIR spectra showed six fundamental absorption bands in the range 500 to 3470 cm⁻¹, whereas the two absorption bands in the range 500-625 cm⁻¹ confirmed the bonding vibrations of metal-oxygen bonds situated at octahedral and tetrahedral sites. UV-Vis spectroscopy was carried out in the wavelength range 200-800 nm and the optical band gap (E_g) was observed in the range 4.2129-4.3115 eV.

Keywords: Nickel chromite nanoparticles; Calcination temperature; Structural properties; Optical properties.

1. Introduction

Mixed spinel metal oxide nanoparticles have attracted substantial attention given their potential applications in quite a lot of areas like ceramics, semiconductors, sensors, and catalytic materials [1-4]. The general formula of spinel structures is AB₂X₄ (X = O, S), and it has a cubic spinel structure and crystal group of Fd3m [5,6]. The spinels are mainly classified into two major variants, namely normal and inverse spinel crystal structures [7,8]. Particularly, transition metal substituted chromites having cubic spinel structure (a class of mixed metal oxides) with formula of MCr₂O₄ (M=Ni, Co, Zn, Mg, etc.) can be described as cubic closely-packed arrangement of oxygen atoms whereas M⁺² and Cr⁺³ ions occupy either tetrahedral (A) or octahedral (B) lattice sites [9]. The unitcell of a spinel structure consists 32 oxygen ions and 24 metal ions. Out of 24 metal ions, 8 metal ions occupy tetrahedral sites and 16 metal ions occupy octahedral sites. They are also known as A-sites and B-sites respectively. NiCr₂O₄ is a normal spinel structure that exhibits ferromagnetic nature. In a normal spinel structure, A-sites are occupied by divalent Ni²⁺ ions and, B-sites are occupied by trivalent Cr³⁺ ions. Enormous applications with this kind of structure have been reported in

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catalytic materials, light and heat-sensitive micromechanical devices, gas sensors, etc. [10-11]. In particular, nickel chromite is acknowledged as a promising catalytic material for various industrial processes [12-15]. Some of the well-developed methods for synthesis of metal oxides are thermal treatment method [16-18], citrate-nitrate gel combustion method [19], sol-gel method [20-22], micro-emulsion method [23], co-precipitation method [24], supersonic radiation method [25], Pechini's method [26], hydrothermal method [27], freeze-drying method [28], and combustion method [29-32]. In the present study, a sol-gel auto-combustion method has been used to synthesize nickel chromite as it produces homogeneous nanoparticles and requires less time of preparation with low cost.

2. Materials and Experimental Procedures

Nickel chromite nanoparticles were synthesized using sol-gel auto combustion method using nickel nitrate [$\text{Ni}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$], chromium nitrate [$\text{Cr}(\text{NO}_3)_3 \cdot 9\text{H}_2\text{O}$] as starting materials in a stoichiometric ratio as shown in Fig. 1.

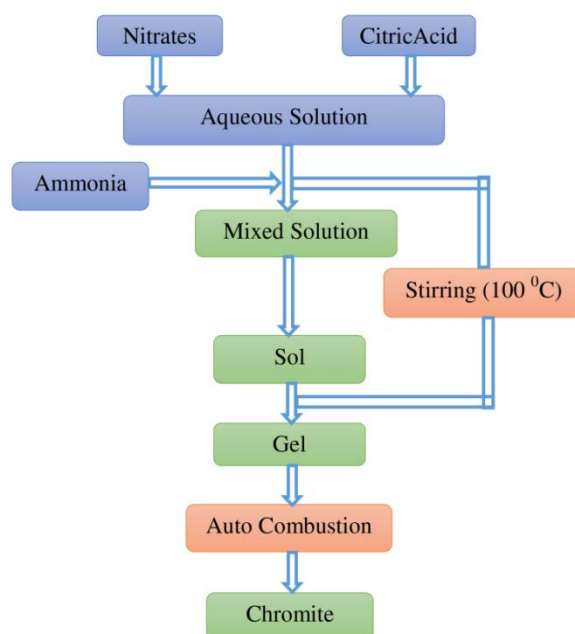


Fig. 1. Flowchart of synthesis of NiCr_2O_4 nanoparticles.

The starting materials in their stoichiometric ratio were dissolved in purified water. The citric acid in a considered quantity was added for initiating a reaction. To maintain pH level 7, ammonia was added drop by drop along with 1 ml of ethylene glycol for de-freezing the solution. The prepared solution was stirred using a magnetic stirrer at about 80°C till it converts into a viscous gel. Thereafter, the precursor was heated at about 300°C for few minutes to get initiated auto combustion. After the auto combustion process, the precursor was made dried and ground into fine powder. The fine powder of nickel chromite (NiCr_2O_4) nanoparticles was divided into four parts and calcined at 700, 900, 1100 and 1300°C for 8 h. The physical parameters were calculated using the data extracted by the powder X-ray diffraction (XRD) using Phillips Expert Diffractometer at room temperature in the 2θ range of 20° - 80° . Microstructural morphological studies and Energy Dispersive X-Ray Spectroscopy (EDS) of the samples were understood by the images obtained by Scanning Electron Microscope (SEM) (ZEISS EVO 18, Special Edition). Fourier Transform Infrared Spectroscopy (FT-IR) was recorded on a Shimadzu FTIR spectrometer (Model: FTIR-8400 S).

within a range of 400-4000 cm^{-1} . UV-Vis spectroscopic studies were recorded by the SYSTRONICS DOUBLE BEAM UV-VIS Spectrophotometer: 2202 in the wavelength range 200-800 nm.

3. Results and Discussion

3.1. X-ray diffraction analysis

The XRD patterns of nickel chromite (NiCr_2O_4) nanoparticles calcinated at 700, 900, 1100 and, 1300°C were carried out using X-ray diffraction technique and presented the same in Fig. 2.

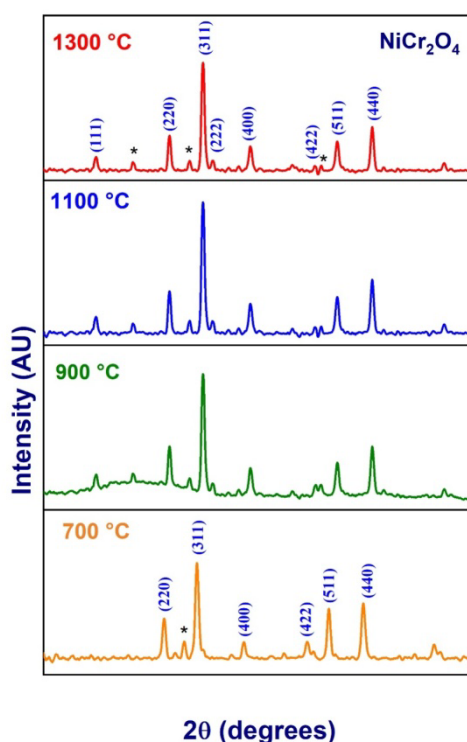


Fig. 2. XRD patterns of NiCr_2O_4 nanoparticles calcinated at 700, 900, 1100 and 1300°C.

From the figure, it is clearly understood that the diffraction pattern consists of well-defined peak sat 18.46° , 29.48° , 30.38° , 35.80° , 37.44° , 43.56° , 54.00° , 57.62° and 63.28° corresponding to reflection planes of (111), (220), (311), (222), (400), (422), (511) and (440) in all the samples except in case of the sample calcinated at 700°C. This is all-clear indication for the formation of spinel structure in all taken up nickel chromite (NiCr_2O_4) samples [33]. In the case of sample calcinated at 700°C, the peak corresponding to (111) peak is missing which can be understood that phase is not fully formed at the calcination temperature of 700°C. Few additional peaks appeared in all the samples which correspond to NiO and they are represented with star (*) symbol. The average crystallite size was calculated using Scherrer formula [34] for all the samples.

$$t = \frac{0.89 \lambda}{\beta \cos \theta} \quad (1)$$

where, t is the average crystallite size, ($\text{\AA}$), λ is the X-ray wavelength (0.15408 nm), θ is the angle of diffraction and β is the full-width at half maximum (FWHM) of the observed peaks. The calculated crystallite size of all the prominent peaks for all the samples is furnished in Table I along with Miller indices of corresponding plane, 2θ , FWHM, interplanar spacing values. The average values of FWHM, crystallite size and interplanar spacing are furnished in Table II. The average crystallite size was increased from 13.73 to 22.33 nm with the increment of calcination temperature from 700 to 1300°C. The lattice parameter was found maximum (8.42 $\text{\AA}$) for the sample calcinated at 700°C and minimum (8.27 $\text{\AA}$) for the sample calcinated at 1100°C. The interplanar spacing was found maximum (2.439 $\text{\AA}$) for the sample calcinated at 1300°C and minimum (2.101 $\text{\AA}$) for the sample calcinated at 700°C.

Tab. I Miller indices, 2θ , FWHM, crystallite size, interplanar spacing of NiCr_2O_4 nanoparticles calcinated at 700, 900, 1100 and 1300°C.

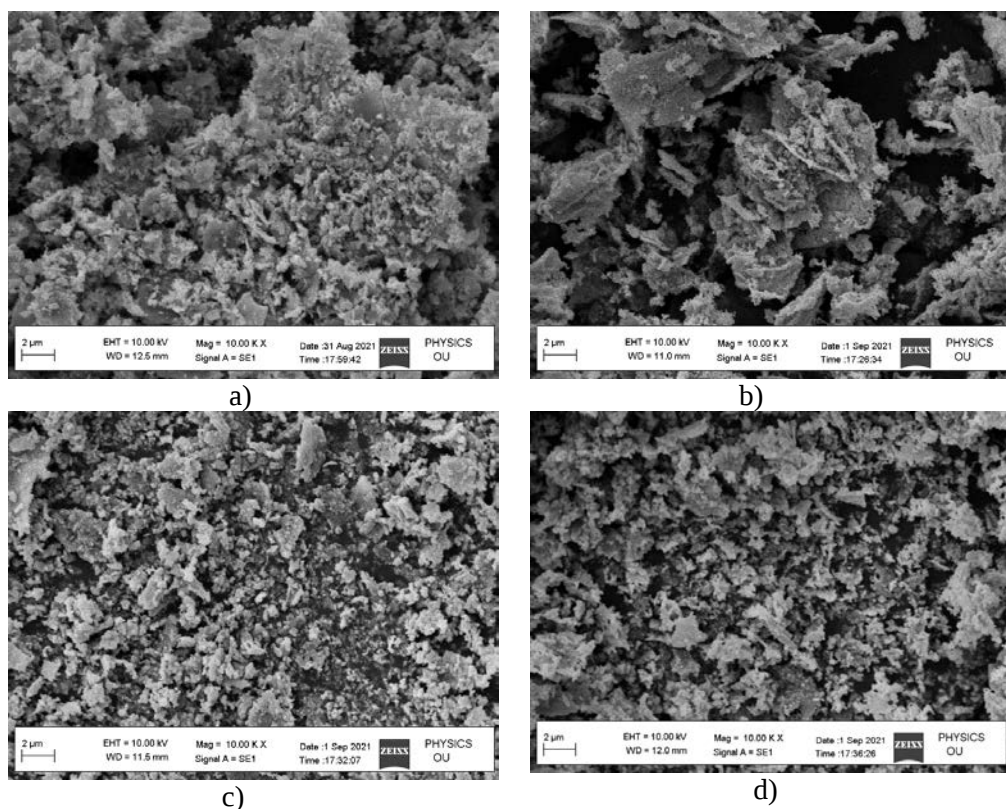
Calcination temperature (°C)	Miller indices (h k l)	2θ (Degrees)	FWHM (Radians)	Crystallite size (nm)	Interplanar spacing (d) ($\text{\AA}$)
700	220	29.48	0.57	14.41415	3.0260
	311	34.78	0.55	15.13855	2.5766
	400	42.36	0.74	11.51509	2.1309
	422	52.72	0.89	9.963435	1.7357
	511	56.20	0.52	17.32130	1.6362
	440	61.74	0.66	14.02523	1.5012
900	111	18.46	0.59	13.64401	4.8116
	220	30.38	0.43	19.14726	2.9404
	311	35.80	0.36	23.19390	2.5069
	222	37.44	0.50	16.77885	2.4021
	400	43.56	0.46	18.60074	2.0787
	422	54.00	0.62	14.38282	1.6970
	511	57.62	0.51	17.77989	1.6003
	440	63.28	0.51	18.29889	1.4700
1100	111	18.46	0.36	22.36102	4.8116
	220	30.38	0.37	22.25223	2.9423
	311	35.80	0.36	23.19390	2.5083
	222	37.46	0.58	14.46538	2.4021
	400	43.56	0.47	18.20498	2.0787
	422	54.00	0.28	31.84768	1.6976
	511	57.60	0.48	18.88932	1.6003
	440	63.22	0.52	17.94120	1.4700
1300	111	18.40	0.36	22.35911	4.8116
	220	30.42	0.39	21.11308	2.9423
	311	35.80	0.34	24.55824	2.5083
	222	37.44	0.42	19.97482	2.4021
	400	43.54	0.46	18.80383	2.0787
	422	54.02	0.27	33.03016	1.6976
	511	57.60	0.45	20.14861	1.6003
	440	63.20	0.50	18.65684	1.4705

Tab. II Average FWHM, average crystallite size, average interplanar spacing, average grain size and lattice parameter of NiCr_2O_4 nanoparticles calcinated at 700, 900, 1100, 1300°C.

Calcination temperature (°C)	Average FWHM (Radians)	Average crystallite size (nm)	Average grain size (nm)	Average interplanar spacing (Å)	Lattice parameter (Å)	Unit Cell Volume (Å ³)
700	0.6550	13.73	140.00	2.101	8.42	597.54
900	0.4975	17.73	141.70	2.438	8.31	573.37
1100	0.4275	21.14	145.52	2.438	8.27	566.66
1300	0.3981	22.33	163.46	2.439	8.30	571.77

3.2. Scanning Electron Microscopy (SEM) Analysis

The morphological study of all the samples was done by scanning electron microscope (SEM). The micrographs of all the nickel chromite (NiAl_2O_4) nanoparticles are presented in Fig. 3. It can be observed that the non-uniform agglomerated fragments have lot of voids and pores which cause the crystallite size lower [35]. It is noticed that all the grains of non-uniform sizes and randomly distributed voids on the surface may bring out greater adsorption and hence better photocatalytic activity [36]. The calculated average grain size of all the samples is furnished in Table II and it was found in the range 140.00 to 163.46 nm. The grain size was increased with calcination temperature. The grain size was found minimum (140.0 nm) for the sample calcinated at 700°C, whereas, the grain size was found maximum (163.46 nm) for the sample calcinated at 1300°C. The bond lengths of at A and B sites are furnished in Table III along with their ionic radii.

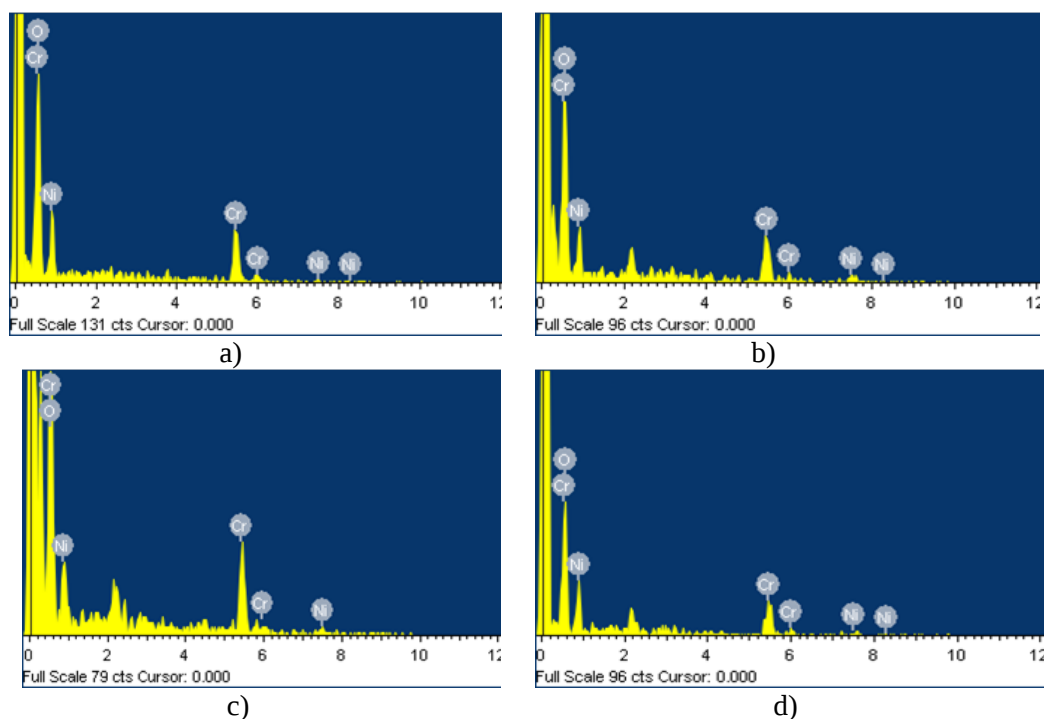
**Fig. 3.** SEM images of NiCr_2O_4 nanoparticles calcinated at 700, 900, 1100 and 1300°C.

Tab. III Bond lengths and Ionic radii in NiCr₂O₄ nanoparticles calcinated at 700, 900, 1100 and 1300°C.

Calcination temperature (°C)	Bond length of A site-O (Å)	Bond length of B site-O (Å)	Ionic radius (r _A) (Å)	Ionic radius (r _B) (Å)
700	1.8235	2.0130	0.3235	0.5130
900	1.7986	2.0769	0.2986	0.5769
1100	1.7915	2.0687	0.2770	0.5687
1300	1.7967	2.0749	0.2967	0.5749

3.3. Energy Dispersive X-ray analysis (EDX)

The existence of NiAl₂O₄ was also confirmed using energy dispersive X-ray analysis (EDX) shown in Fig. 4 and the percentage of atomic weights are furnished in Table IV. It revealed Ni, Cr, and O peaks and indicated the existence of NiAl₂O₄ phase without any other impurity [37]. But the nickel atomic weights have appeared more in samples calcinated at temperatures 700 and 1300°C, which reveals that the NiO fraction is more compared to other samples. It is all in very good agreement with the XRD and FTIR investigation. It was observed that at 700°C the phase was not fully formed and NiO fraction appeared more and at higher temperature 1300°C, crystallization was done properly and phase was formed perfectly, but the NiO fraction was appeared more. Hence, it can be concluded that the formation of nickel chromite (NiCr₂O₄) nanoparticles with perfect phase formation and NiO elimination could be done having the calcination in the temperature range 900-1100°C.

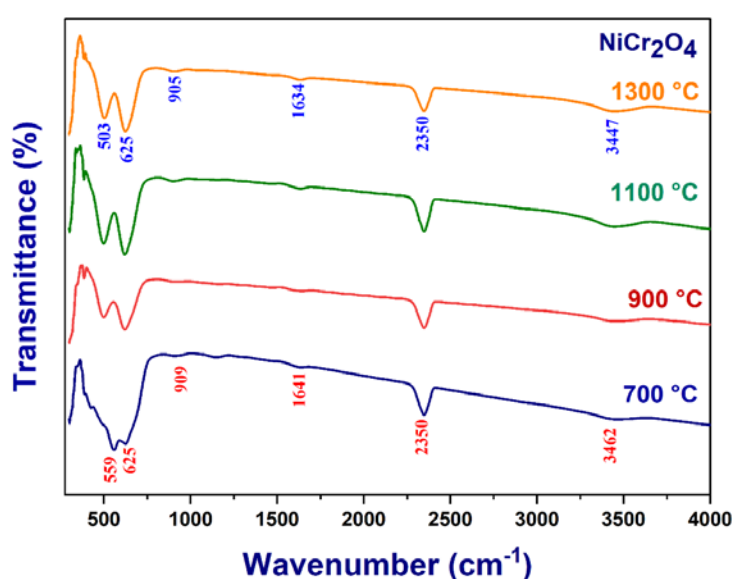
**Fig. 4.** EDS patterns of NiCr₂O₄ nanoparticles calcinated at 700, 900, 1100 and 1300°C.

Tab. IV Elemental percentage in NiCr₂O₄ nanoparticles calcinated at 700, 900, 1100 and 1300°C.

Calcination temperature (°C)	Element	Atomic %
700	Ni	20.28
	Cr	19.14
	O	60.59
900	Ni	14.24
	Cr	41.71
	O	44.06
1100	Ni	13.32
	Cr	26.44
	O	60.24
1300	Ni	23.76
	Cr	16.14
	O	60.10

3.4. Fourier transform infrared (FT-IR) analysis

The FTIR spectra of nickel chromite (NiAl₂O₄) samples are shown in Fig. 5 and the characteristic frequencies are presented in Table V. FTIR analysis was used to identify the vibrational stretch frequency of metal-oxygen bonds [38]. All spectra exhibited bands in the regions 3440-3462 cm⁻¹ and 1634-1641 cm⁻¹ which represent the -OH stretching vibrations and deformative vibration of water molecules respectively [39]. Another band was observed in the region 2345-2350 cm⁻¹ which can be accredited to the vibrations of carbon impurity atoms which resemble the stretching of Si-C bonding [40]. It was observed that the band in the region 899-918 cm⁻¹ corresponds to the C-C stretching and bending vibrations. The metal-oxygen stretching bands in the range 619-625 cm⁻¹ and 500-560 cm⁻¹ can be associated with the vibrations of the tetrahedrally and octahedrally coordinated Cr-O bond and the octahedrally coordinated Ni-O, respectively. This characteristic was identified in the formation of zinc chromite spinel structure [41-42].

**Fig. 5.** FTIR spectra of NiCr₂O₄ nanoparticles calcinated at 700, 900, 1100 and 1300°C.

Tab. V Characteristic wavenumbers of FTIR spectra of NiAl₂O₄ nanoparticles calcinated at 700, 900, 1100 and 1300°C.

Calcination temperature (°C)	ν_1 (cm ⁻¹)	ν_2 (cm ⁻¹)	ν_3 (cm ⁻¹)	ν_4 (cm ⁻¹)	ν_5 (cm ⁻¹)	ν_6 (cm ⁻¹)
700	559	625	909	1641	2350	3462
900	500	621	918	1641	2345	3440
1100	500	619	899	1636	2345	3440
1300	503	625	905	1634	2350	3447

3.5. UV-Visible Spectroscopy

The UV-visible optical absorption spectra of the nickel chromite nanoparticles calcinated at 700, 900, 1100 and 1300°C were taken up to study their optical properties. The absorption spectra of all the samples were carried out using UV-visible spectrophotometer (model: Lambda 35, maker: Perkin Elmer) in the wavelength range 200-800 nm using quartz cuvettes at room temperature. Halogen and deuterium lamp are used as sources for visible and UV-radiations respectively at room temperature. UV-visible absorption spectroscopy is a powerful technique to explore the optical properties of semiconducting nanoparticles [43]. The UV-visible optical absorption spectra of all the samples are presented in Fig. 6.

Tab. VI Band gap of NiCr₂O₄ nanoparticles calcinated at 700, 900, 1100 and 1300°C.

Calcination Temperature (°C)	Band gap (E _g) (eV)
700	4.2203
900	4.1143
1100	4.0896
1300	3.9223

The absorption would take place due to several aspects, such as band gap, surface roughness, impurity centers and oxygen deficiency. The absorption spectra showed that the absorption is increased with increasing calcination temperature, and exhibits an absorption edge at around 383-379 nm, which can be ascribed to the photo excitation of electrons from valence band to conduction band [44]. The absorption edge of different samples slightly varies as the calcination temperature varies. The observed slight shift in the samples is due to the formation of nickel chromite spinel phase (NiCr₂O₄). The optical absorption coefficient α is evaluated using the relation [45].

$$\text{Absorption coefficient } (\alpha) = A/d \quad (2)$$

where d is known as the thickness of sample cell and A is known as the absorbance. The optical band gap is evaluated using the Tauc relation [46].

$$(\alpha h\nu) = A (h\nu - E_g)^{1/2} \quad (3)$$

where α is known as the absorption coefficient, $h\nu$ is known as the incident photon energy, A is known as proportional constant and E_g is known as the optical band gap of the material. According to equations (2) and (3), by taking extrapolating the linear part of a curve of α^2 versus photon energy ($h\nu$) to the abscissa, the energy band gap can be derived. The estimated optical band gap (E_g) value for all the samples was calculated using Tauc plots. The optical

band gap was found in the range 3.9223–4.2203 eV and the same furnished in Table VI. It can be observed that the optical band gap was found minimum ($E_g = 3.9223$ eV) for the sample calcinated at 1300°C, further it was decreased with calcination temperature and found maximum (4.2203 eV) for the sample calcinated at 700°C. The energy gap (E_g) values were found to decrease with increasing calcination temperature. This decrease of E_g can be accredited to the encouragement of defects levels with the calcination temperature. Therefore, the NiCr_2O_4 calcinated at lower temperature can absorb more photons and generate more electron and holes, which is favorable for a higher photocatalytic activity compared to the NiCr_2O_4 calcinated at higher temperature.

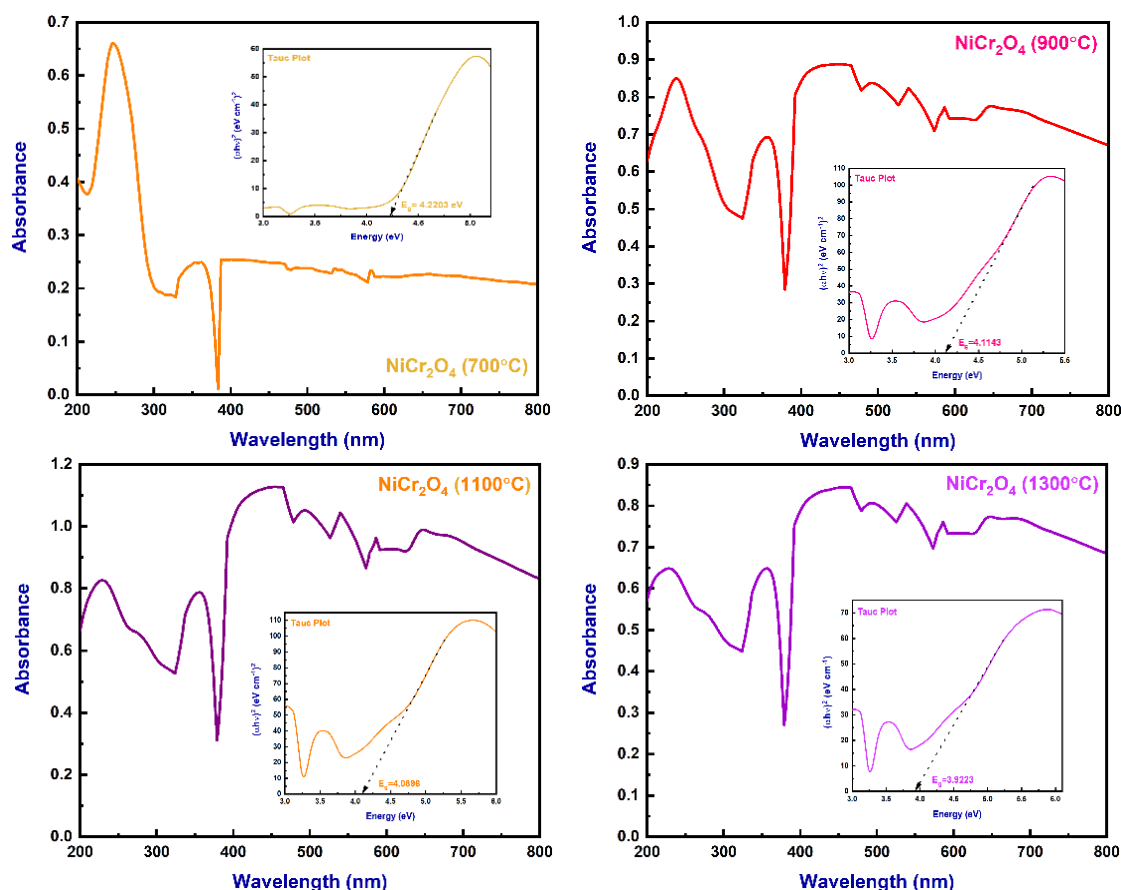


Fig. 6. UV-Vis spectra with Tauc plot (Inside) of NiCr_2O_4 nanoparticles calcinated at 700, 900, 1100 and 1300°C.

4. Conclusion

Nickel chromite (NiCr_2O_4) nanoparticles were synthesized by sol-gel auto combustion method successfully. The prepared powder is divided into four parts and calcinated at 700, 900, 1100 and 1300°C. X-ray diffraction patterns confirmed that all the samples have spinel structure with space group $\text{Fd}\bar{3}\text{m}$. The crystallite size was found to increase from 13.73 to 22.33 nm with calcination temperature. The average grain size was found increased with calcination temperature and found in the range 140.00 to 163.46 nm. The lattice parameter was found in the range of 8.27 to 8.42 Å where as the interplanar spacing was found in the range 2.101 to 2.439 Å. The SEM images showed non-uniform agglomerated fragments having lot of voids and pores. FTIR spectra showed fundamental six

absorption bands in the range 500 to 3450 cm^{-1} . The first two absorption bands in the range 500-625 cm^{-1} related to the bonding vibrations of Cr-O, Ni-O situated at octahedral and tetrahedral sites. UV-Vis spectroscopy was carried out in the wavelength range 200-800 nm and the optical band gap (E_g) was observed in the range 3.9223-4.2203 eV.

Acknowledgments

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Сажетак: Наночестице никл-хромита су припремљене сол-гел ауто сагоревајућом методом уз додатну калцинацију на различитим температурама, 700, 900, 1100 и 1300°C током 8h. Припремљене наночестице су карактерисане методама XRD, SEM,

EDS, FTIR и UV-Vis спектроскопијом. Из XRD шема, Милерови индекси ($h\ k\ l$) су утврђени за све пикове, и било је јасно да једињење кристализује према спинелној структури групе $Fd3m$. SEM микрографије указују на не-униформне агрегираних фрагменте са доста пора. Употребом Шерерове методе, израчуната је величина кристалита и утврђено је да расте са 13.73-22.33 nm до 140.00-163.46 nm са температуром калцинације. FTIR спектри су показали шест основних трака у опсегу 500-3470 cm^{-1} где су две траке у рангу 500-625 cm^{-1} потврдиле вибрације метал-оксид везе. UV-Vis спектроскопија је изведена у таласном опсегу 200-800 nm и оптички процеп (E_g) је примећен у рангу 4.2129-4.3115 eV.

Кључне речи: наночестице никл-хромита; температура калцинације; структурна својства; оптичка својства.

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