STRUCTURAL PROPERTIES OF NiO AND CuO NANOPARTICLES AND NiO/CuO NANOCOMPOSITE SYNTHESIZED VIA FACILE PRECIPITATION PROCESS

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Abstract : **We report the synthesis of high quality NiO and CuO nanoparticles and NiO/CuO binary metal oxide nanocomposites were synthesized by a simple chemical precipitation process. Structural and sizes of the synthesized product was studied by powder X-ray diffraction (XRD) pattern. The XRD pattern proves that the final product has cubic-monoclinic phases of NiO/CuO nanocomposites, XRD results also indicated that the crystalline properties of the nanocomposite were improved without affecting the parent lattice**.

Keywords: Metal oxide; Metal composites; Structural properties

I. INTRODUCTION

Metal oxides play a very important role in many areas of chemistry, physics and materials science. In the emerging field of nanotechnology, a goal is to make nanostructures or nanoarrays with special properties with respect to those of bulk or single particle species. Oxide nanoparticles can exhibit unique physical and chemical properties due to their limited size and a high density of corner or edge surface sites. Particle sizes in the nano and specific crystal morphologies are expected to enhance the performance and allow the fine tuning of the properties of these materials. The combination of two or more metals in an oxide matrix can produce materials with novel physical and chemical properties leading to relatively higher performance in various technological applications. During the last few years, synthesis of metal oxide nanocomposite materials have been attracted considerable attention [1–5]. Nickel oxide (NiO) is an attractive material due to its excellent chemical stability, as well as optical, electrical and magnetic properties. Furthermore, it is considered to be a model semiconductor with p-type conductivity films due to its wide band-gap energy range from 3.6 to 4.0 eV [6]. Copper oxide (CuO) is one of potential p-type semiconductors and gains considerable attentions due to its excellent optical, electrical, physical, and magnetic properties. CuO with narrow band gap of 1.2 eV is extensively used in various applications [7, 8]. These two oxides have been widely used in almost the same application areas. Developing a new composite material by combining them into one could open up a new direction for research and applications. With this motivation, NiO and CuO nanoparticles and NiO/CuO nanocomposites were prepared by simple precipitation process. The as-synthesized samples are subjected to the powder X-Ray Diffraction (XRD) analysis and their structural and sizes were compared.

II. EXPERIMENTAL PROCEDURE

Synthesis of NiO/CuO nanocomposites

The nickel oxide/copper oxide nanocomposites were prepared by the facile precipitation process. All the chemical reagents were commercial with AR purity, and used directly without further purification. In a typical experiment, 0.1M of nickel (II) acetate tetrahydrate ($Ni(CH_3COO)_2{\cdot}4H_2O$) was dissolved in 100 ml distilled water. The precipitates were obtained by the addition of 0.2 M of sodium hydroxide (NaOH) pellets to the above solution, which was stirred for one hour. The resultant precipitate was filtered, washed with distilled water and absolute ethanol to remove the impurities, and dried at 120ºC for 15 hrs. Then, light greenish colored NiO sample was obtained, when dried sample was calcined at 450ºC for 2h. The same procedure was followed for the preparation of CuO sample. For the preparation of NiO/CuO nanocomposite, 0.1M of nickel acetate tetrahydrate and copper acetate monohydrate were dissolved in 100 ml distilled water. The precipitates were obtained by the addition of 0.4 M of sodium hydroxide (NaOH) pellets to the above solution, which was stirred for one hour. Further, dried and calcinations process were same procedure followed for preparing NiO and CuO samples.

The formation of NiO, CuO nanoparticles and NiO/CuO nanocomposites is given in the equation below:

$$Ni(CH_3COO)_2{\cdot}4H_2O + 2NaOH \rightarrow Ni(OH)_2 + 2(NaCH_3COO) + 4H_2O$$
$$Ni(OH)_2 \rightarrow NiO + H_2O \quad \text{---------- (1)}$$

$$Cu(CH_3COO)_2{\cdot}H_2O + 2NaOH \rightarrow Cu(OH)_2 + 2(NaCH_3COO) + H_2O$$
$$Cu(OH)_2 \rightarrow CuO + H_2O \quad \text{---------- (2)}$$

$$\left.\begin{matrix} Ni(CH_3COO)_2{\cdot}4H2O \\ + \\ Cu(CH_3COO)_2{\cdot}H_2O \end{matrix}\right\} + 4NaOH \rightarrow Ni(OH)_2/Cu(OH)_2 + 4(NaCH_3COO) + 5H_2O$$
$$Ni(OH)_2/Cu(OH)_2 \rightarrow NiO/CuO + 2H_2O \quad \text{---------- (3)}$$

Characterization of synthesized nanocomposites

The characterization of metal oxide nanocomposites is essential for understanding of their structural and particles sizes. Due to the inherent difficulties involved, the scientific experiments for the characterization should have the ability for rapid collection of data of several parameters with good precision and accuracy. The development of novel tools and instruments is one of the greater challenges in nanotechnology. The crystalline size and structures of the prepared NiO, CuO and NiO/CuO samples were characterized by X-ray diffraction on a rotating-target X–ray diffractometer (JSO-DEBYFLEX 2002) equipped with monochromatic high intensity CuKα1 radiation (λ = 0.15406 nm, 40 kV, 100 mA). The samples were scanned in the range of 20 to 70° (2θ), at a scanning rate of 0.005°/s and step size of 0.02°.

III. Results and Discussion

X-ray diffraction (XRD) is a rapid analytical technique primarily used for the phase identification of a crystalline material, and can provide information on unit cell dimensions. This method uses a monochromatic source of X-rays and measures the pattern of diffracted radiation, which is a result of the constructive interference due to the crystalline structure of the powder. The crystallite size can be obtained either by direct computer simulation of the X-ray diffraction pattern or from the Full Width at Half Maximum (FWHM) of the diffraction peaks using the Debye-Scherrer's formula [9].

$D=0.9\lambda/\beta\cos\theta$

where,

λ - Wavelength of X-rays,
β - FWHM in radian,
θ - Peak angle.

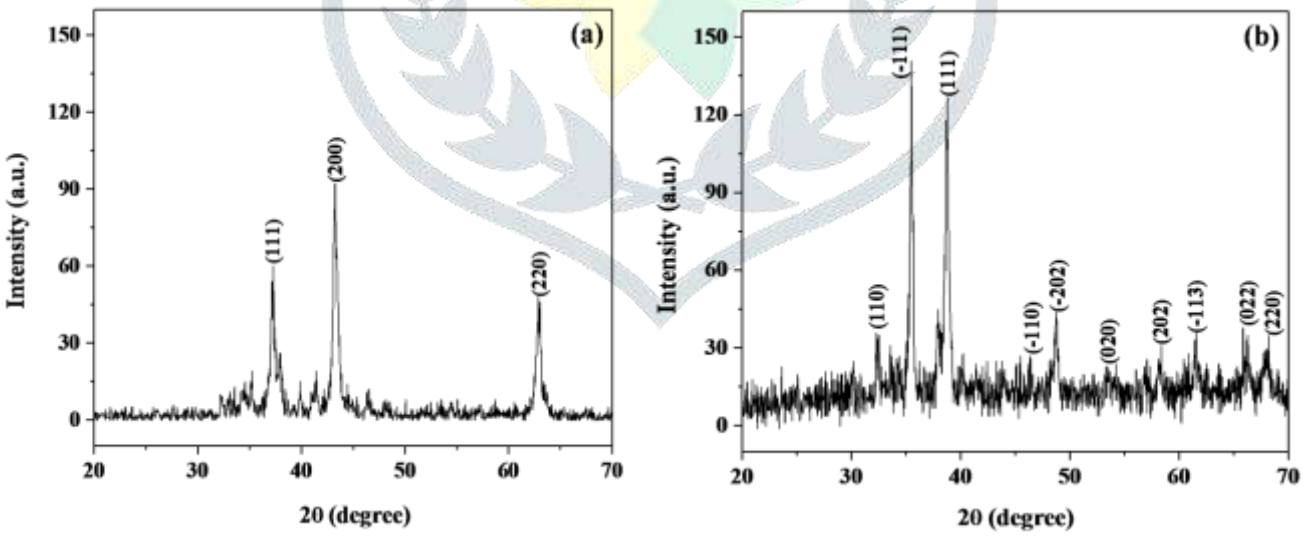

Fig. 1 XRD patterns of (a) NiO and (b) CuO nanoparticles.

It can be seen from Fig. 1(a) that the diffraction peaks are low and broad due to the small size effect and incomplete inner structure of the particle. The peaks positions appearing at 2θ are 37.36°, 43.28° and 63.98° can be readily indexed as (111), (200) and (220) crystal planes of the bulk NiO, respectively. All these diffraction peaks can be perfectly indexed to the face-centered cubic (FCC) crystalline structure of NiO, not only in peak position, but also in their relative intensity of the characteristic peaks, which is in accordance with that of the standard spectrum (JCPDS, No. 04-0835). The XRD pattern shows that the samples are single phase and no any other impurities distinct diffraction peak except the characteristic peaks of FCC phase NiO was detected. This result shows that the physical phases of the NiO nanoparticles have higher purity prepared in this work [10].

The XRD pattern of synthesized CuO nanoparticles is shown in Fig.1 (b). XRD peaks confirm that the formation of CuO was in monoclinic phase. The predominant characteristic peaks located at 2θ are 35.49°, 38.77° and 48.81° are assigned to (-111), (111) and (-202) plane orientation of CuO (JCPDS 80-1268). The details of the 2θ degree, FWHM (by Gaussian fit), peak intensity, crystalline size of each peak and average crystalline size of the NiO and CuO nanoparticles were given in Table 1. From the analysis, compared with CuO nanoparticles, NiO nanoparticles shows lower intensities of peak and high FWHM values with low crystalline size.

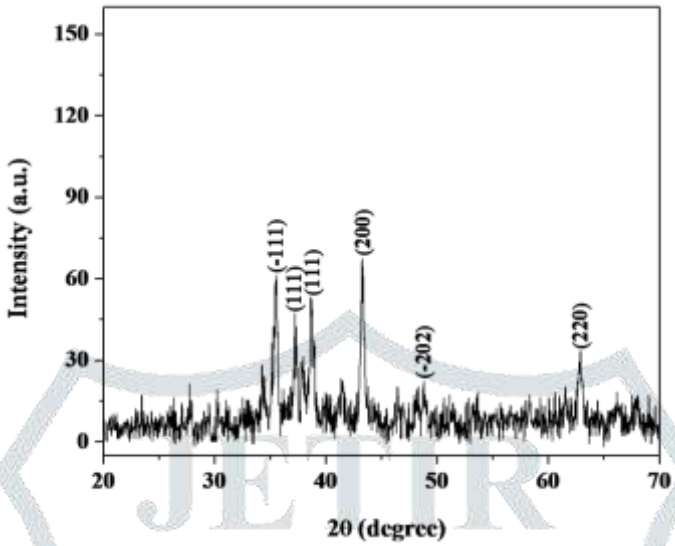

Fig. 2 XRD pattern of NiO/CuO nanocomposites.

Name of the sample	2θ (degree)	FWHM	Intensity of the peak (a.u.)	Calculated crystalline size (nm)	Average crystalline size (nm)
NiO	37.36	0.24304	59.92	36.05	30.46
	43.28	0.33453	91.81	26.68	
	62.98	0.33945	47.93	28.66	
CuO	35.49	0.27054	140.52	32.22	31.12
	38.77	0.29449	126.7	29.88	
	48.81	0.29139	43.44	31.28	

Table 1 Details of 2θ (degree), FWHM, intensity of the peak, calculated crystalline size of each peak and average crystalline size of the NiO and CuO nanoparticles.

Name of the sample	2θ (degree)	FWHM	Intensity of the peak (a.u.)	Calculated crystalline size (nm)	Average crystalline size (nm)
NiO/CuO	35.48	0.29013	61.17	30.04	23.62
	37.24	0.43907	46.82	19.95	
	38.7	0.37047	53.11	23.75	
	43.26	0.45301	67.08	19.71	
	48.68	0.31301	22.01	29.11	
	62.86	0.50659	33.03	19.2	

Table 1 Details of 2θ (degree), FWHM, intensity of the peak, calculated crystalline size of each peak and average crystalline size of the NiO/CuO nanocomposites.

Fig.2 shows the X-ray diffraction (XRD) pattern of the NiO/CuO Nanocomposites. As seen in Fig.2 , the predominant characteristic peaks were obtained at 2θ values of 37.24°, 43.26° and 62.86° correspond to planes of as (111), (200) and (220) related to cubic structure of NiO and other 2θ values of 35.48°, 38.7° and 48.68° correspond to planes of as (-111), (111) and (-202) related to monoclinic structure of CuO, this fact indicates that the prepared sample is not a single phase but a composite. Moreover, no impurity such as $Ni(CH_3COO)_2$, $Ni(OH)_2$, $Cu(CH_3COO)_2$ and $Cu(OH)_2$ were detected. The details of 2θ degree, FWHM (by Gaussian fit), peak intensity, crystalline size of each peak and average crystalline size of the NiO/CuO nanocomposites were given in Table 2. From the tables it was concluded that the composite of NiO/CuO samples shows greater

effect to reduction in particles size as compared with single phase of metal oxide nanoparticles. Hence, it should be suitable for various applications compared with single phase of NiO and CuO nanoparticles.

IV. CONCLUSION

In summary, the simple synthesis method has been successfully proposed represents an interesting approach to produce NiO and CuO nanoparticles and NiO/CuO nanocomposites. The cubic and monoclinic structure of NiO and CuO nanoparticles and cubic/monoclinic structure of NiO/CuO nanocomposites were confirmed by the powder XRD patterns and the average particle size of the samples calculated to be 30.46, 31.12 and 23.62 nm. Further, the proposed method has the important advantage of being simple, fast, and cost effective and the prepared lower crystalline size of the NiO/CuO nanocomposites are promising good candidate for numerous applications.

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