Malaysian Journal of Catalysis
http://mjcat.utm.my/
MJCat
Research Article

Fabrication and characterization of $(Cr_2O_3)_6$ $(CdO)_4$ nanoparticles produced utilizing calcination temperature route

Naif Mohammed Al-Hada[1,2*], Rafiziana Md. Kasmani[2*], Abbas M. Al-Ghaili[3*], Hussein Baqiah[1], Muneer Aziz Saleh[4], Hairoladenan Kasim[5], M. N. Azlan[6]

[1]*Shandong Key Laboratory of Biophysics, Institute of Biophysics, Dezhou University, Dezhou 253023, China*
[2]*School of Chemical and Energy Engineering, Universiti Teknologi Malaysia, 81310 Skudai, Johor Bahru, Johor, Malaysia*
[3]*Institute of Informatics and Computing in Energy (IICE), Universiti Tenaga Nasional (UNITEN), 43000 Kajang, Selangor, Malaysia*
[3] *Office of Radiation Protection, Environmental Public Health, Washington State Department of Health*
[5]*College of Computing & Informatics (CCI), Universiti Tenaga Nasional (UNITEN), 43000 Kajang, Selangor, Malaysia*
[6]*Physics Department, Faculty of Science and Mathematics, University Pendidikan Sultan Idris, Tanjong Malim 35900, Malaysia*

**Corresponding Author: naifalhada@yahoo.com; rafiziana@utm.my;abbasghaili@yahoo.com*

Article history :
Received 1 October 2021
Accepted 30 January 2022

ABSTRACT

A unique thermal calcination temperature was used to produce $(Cr_2O_3)_{0.6}$ $(CdO)_{0.4}$ nanoparticles. X-ray diffraction (XRD), energy-dispersive X-ray spectroscopy (EDX), and field emission scanning microscopy were used to characterize the structural, morphological, and elemental composition aspects of the generated nanoparticles (FESEM). The XRD analysis demonstrated that the crystalline size has been increased after calcination. The crystalline size rose with increasing calcination temperature, and the mean grain size investigated by FESEM micrographs revealed a matching growing tendency. The existence of Cr, Cd, and O in these new compounds has been confirmed by EDX studies.

Keywords: Heat treatment; Polyvinylpyrrolidone; $(Cr_2O_3)_{0.6}$ $(CdO)_{0.4}$ nanoparticles

| eISSN 0128-2581 |

1. INTRODUCTION

Recently, the promising characteristics of semiconductor nanostructured materials have been exploited by many applications, where this has increased research's interests [1-3]. One of the most used applications is the hexagonal chromium oxide (Cr_2O_3) and cubic cadmium oxide (CdO), n-type II–VI semiconductors [4, 5]. Given that both materials have shown their capacity in gas sensing, and its mixed form i.e., Cr_2O_3-CdO might be considered as a useful material in applications such as for the photovoltaic industry [6]. Another property of mixed Cr_2O_3 and CdO might also display complementary characteristics; for example: display multiple band gaps from both oxide semiconductors that may possess significant properties from individual semiconductor components [7, 8]. However, this study is not common practise as it tries to develop a product that can fit for industrial uses, focusing on simple handling and particle sizes of varying sizes. Smaller sizes can be employed for many uses, whereas larger sizes can be used for energy applications. Other advantages include reduced cost, good quality, high flexibility, powdered form, and an effective band gap. Notably, the current approach does not necessitate the use of any extra chemical reagents. This research focuses on a unique technique for manufacturing a $(Cr_2O_3)_{0.6}$ $(CdO)_{0.4}$ nanosize and the effect of temperature [9-25].

This study describes a simple calcination temperature process to produce $(Cr_2O_3)_6$ $(CdO)_4$ nanoparticles. An overview related to producing pure nanoparticles is provided. Pure nanoparticle production is a solution containing nitrate metallic ions and PVP capping agent followed by calcination at a required temperature before undergoing structural, morphological, and optical characteristics investigation.

2. EXPERIMENTS

2.1 Materials

Chromium nitrate $Cr(NO_3)_3{\cdot}6H_2O$ and cadmium nitrate $Cd(NO_3)_2.4H_2O$ metallic salts were employed as metal precursors, polyvinyl pyrrolidone (PVP) was used as a capping agent for facilitating the dispersion of nanoparticles, and deionized water was used as a solvent. $Cr(NO_3)_3{\cdot}6H_2O$ (99%), $Cd(NO_3)_2.4H_2O$ (99%) and PVP (MW= 58,000) were procured from Sigma-Aldrich.

2.2 Method

Following the dissolution of 0.6 mmol of $Cr(NO_3)_3{\cdot}6H_2O$, 0.4mmol of $Cd(NO_3)_2.4H_2O$, and 2% PVP have been added completely to form a homogeneous solution. The resulting solution has been poured into a Petri dish and dried at a temperature of 363K for 24 h producing thereby a yellow-colored dried gel which was crushed into powder and calcined at temperatures 773, 873, 973, and 1073K. for 3 h. To determine the structural, morphological, elemental composition, and optical properties of the $(Cr_2O_3)_{0.6}$ $(CdO)_{0.4}$ nanoparticles, various analytical instruments were employed. This included powder X-ray diffraction (XRD), field emission scanning electron microscopy (FESEM), and energy-dispersive X-ray spectroscopy (EDX).

3. RESULTS AND DISCUSSION

Fig. 1 displays the XRD patterns for the samples calcined at 773, 873, 973, and 1073 K. The reflections observed for the synthesized powder confirm the existence of the hexagonal phase of Cr_2O_3 a nd cubic phase of CdO semiconductor nanoparticles. A mixture of hexagonal Cr_2O_3 semiconductor nanoparticles and cubic phase CdO semiconductor nanoparticles was displayed by the synthesized $(Cr_2O_3)_{0.6}$ $(CdO)_{0.4}$ nanoparticles. The position of the Bragg's lines of the Cr_2O_3 nanoparticles with reference to JCPDS Card 38-1479 [22], the presence of multiple diffraction peaks of (012), (104), (110) (211), and (030) in the diffraction patterns suggests that the Cr_2O_3 sample displays a characteristic hexagonal structure. Similarly, the position of Bragg's lines of CdO nanoparticles was used to determine the interplanar spacing (d), which in turn used to index the diffraction peaks. The existence of multiple diffraction peaks of (200), (220), (311), and (222) in the diffraction patterns suggests that the CdO samples have a typical face centered cubic (fcc) structure referring to the PDF Card no. 005- 0640 data [26].

The full width of the half-maximum peak broadening of the peak of the XRD patterns was employed to calculate the average crystal size, using the Scherer formula:

$$D=0.9\lambda/\beta\cos\theta \quad (1)$$

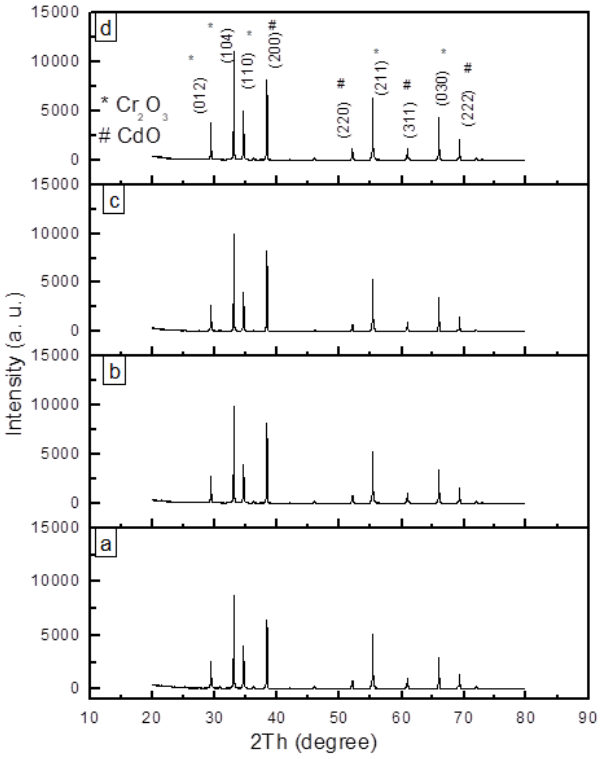

Fig. 1. XRD patterns of binary $(Cr_2O_3)_{0.6}$ $(CdO)_{0.4}$ nanoparticles prepared at various calcination temperatures: (a) 773, (b) 873, (c) 973, (d) 1073K.

Where D is the crystalline size (nm), β is the entire width of the diffraction line at half of the maximum intensity measured in radians, λ is the X-ray wavelength of Cu Kα = 0.154 nm and θ is the Bragg's angle [27]. The results displayed that the increases in crystal sizes were determined using the Scherer formula at calcination temperatures ranging from 773 -1073K as shown in Table 1. As the calcination temperature increased, a corresponding increase in the crystal size w, which is ascribed to the enlargement of grain size [28]. It is concluded from Fig. 1 (a-d) that sharper and narrower diffraction peaks with increased intensities result when the calcination temperature is raised pointing to a significant enhancement in the crystallinity.

Table 1. The crystalline size of the sample's nanoparticles calcined at various temperatures.

Calcination temperature (K)	Crystal size, D_{XRD} (nm)
773	59
873	71
973	85
1073	92

Field emission scanning electron microscopy (FESEM) in the range of 773-1073K has been used to study the surface morphology of the sample nanoparticles. Micrographs of $(Cr_2O_3)_{0.6}$ $(CdO)_{0.4}$ nanoparticles, at various calcination temperatures (773, 873, 973, and 1073 K), are displayed in Fig. 2. The Cr_2O_3 and CdO compounds are seen as homogenous. At low temperatures (Fig. 2 (a-b)), the grain size of the sample is small and virtually rod in shape with some regularities. In Fig. 2 (c-d) the grain size of sample increase and overlap (fuse together to form larger particle sizes by melting their surfaces) with increasing calcination temperature. As that the calcination temperature increased, the particles increased in size individually, resulting in a more even distribution and consistency throughout the sample (Figure 4c and Figure 4d). In order to calculate the average grain size of the sample, Image J software was used to measure the grain size of around 70 grains in a FESEM image. At temperatures of 773, 873, 973, and 1073 K, the average grain sizes are 1.84, 2.10, 2.30, and 2.55 microns, respectively.

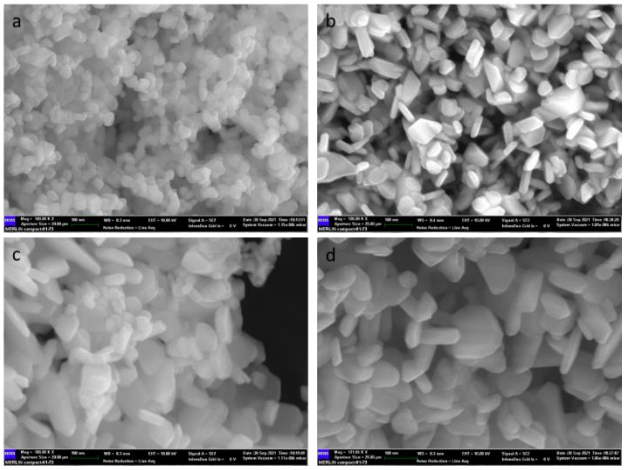

Fig. 2. FESEM images of $(Cr_2O_3)_{0.6}$ $(CdO)_{0.4}$ nanoparticles prepared at various calcination temperatures: (a) 773 (b) 873, (c) 973 and (d) 1073 K.

The EDX spectra and atomic composition of $(Cr_2O_3)_{0.6}$ $(CdO)_{0.4}$ nanoparticles formed at 873K by the calcination route are explained in Fig. 3. The fabrication of $(Cr_2O_3)_{0.6}$ $(CdO)_{0.4}$ nanoparticles has been certified by the corresponding peaks of Cr, Cd, and O produced in the sputtered sample. A 6:4 ratio of Cr to Cd precursors, correlating with their composition in the chemical formula $(Cr_2O_3)_{0.6}$ $(CdO)_{0.4}$ nanoparticles, was determined by the atomic composition (%) ratios of [Cr:O] and [Cd:O], shown to be [28: 32] % and [19: 21] % respectively. The sputtering process applied during the preparation of the sample for EDX analysis generated the peaks of Pt as seen in the spectrum. The success of the calcination employed in this work has been validated by the lack of element loss seen during the fabrication process, and the fabrication of $(Cr_2O_3)_{0.6}$ $(CdO)_{0.4}$nanoparticles as confirmed by the EDX spectrum data.

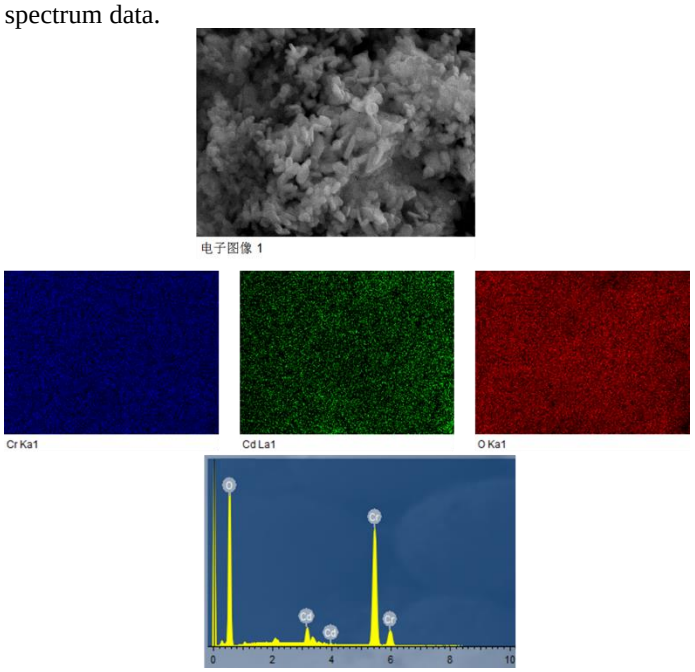

Fig. 3. Shows the EDX spectrum of the $(Cr2O3)_{0.4}(CdO)_{0.6}$ nanoparticles calcined at 873 K.

4. CONCLUSION

As proven by XRD examinations of $(Cr_2O_3)_{0.}6$ $(CdO)_{0.4}$ nanoparticles produced by the novel calcination approach, the synthesis of $(Cr_2O_3)_{0.}6$ $(CdO)_{0.4}$ nanoparticles with hexagonal and face-cantered cubic structures, respectively, was achieved at all calcination temperatures investigated. The size of the crystal was demonstrated to increase with higher calcination temperatures, as demostrated by the crystal diameters, increasing from 59 nm at 773K to 92 nm at 1073K, respectively. Further, the EDX studies shown that the peaks of Cr and Cd, an atomic composition consisting primarily of chromium was found and it was in agreement with the quantities of Cr and Cd used in the initial materials. The lack of element loss seen during the synthesis process, as well as the successful creation of $(Cr_2O_3)_{0.}6$ $(CdO)_{0.4}$ nanoparticles, demonstrated the efficiency of the thermal treatment method used in this study.

ACKNOWLEDGEMENTS

This work was supported in part by the Research Foundation for Advanced Talents of Dezhou University, in part by Universiti Teknologi Malaysia (UTM), Fellow Research Grant, Q.J130000.21A6.00P19 and MOHE- FRGS grant (R.J130000.7851.5F392).

REFERENCES

[1] L. Zhu, M. Gao, C. K. N. Peh, and G. W. Ho, Materials Horizons, 5 (2018) 323-343.
[2] F. Cansell and C. Aymonier, The Journal of Supercritical Fluids, 47 (2009) 508-516.
[3] J. H. Kim et al., Advanced Functional Materials, 30 (2020) 1902049.
[4] N. M. Al-Hada et al., Journal of Materials Research and Technology, 11 (2021) 252-263.
[5] C. Karakaya, Bilkent University, 2012.
[6] M. Gong, Stanford University, 2016.
[7] N. M. Noah, Journal of Nanomaterials, 2020 (2020).
[8] M. Vargas, The University of Texas at El Paso, 2015.
[9] E. Absi, M. Saleh, N. M. Al-Hada, K. Hamzah, A. M. Alhawsawi, and E. M. Banoqitah, Applied Physics A, 127 (2021) 1-32.
[10] N. M. Al-Hada et al., Journal of Materials Research and Technology, 11 (2021) 252-263.
[11] N. M. Al-Hada et al., IEEE Access, 8 (2020) 93444-93454.
[12] N. M. Al-Hada et al., IEEE Access, (2020).
[13] N. M. Al-Hada et al., International Journal of Nanomedicine, 12 (2017) 8309.