doi:10.1017/S1431927622010066
Microsc. Microanal. 28 (Suppl 1), 2022

Characterization of Electrospun $ZnCr_2O_4$ Spinel Nanofibers

Menuka Adhikari, Tamela L. Moore, Zamari T. Morris, Shubo Han and Zhiping Luo*

Department of Chemistry, Physics and Materials Science, Fayetteville State University, Fayetteville, North Carolina, United States.
* Corresponding author: zluo@uncfsu.edu

Spinel oxides with a general formula of AB_2O_4, where A is usually occupied by a divalent cation such as Mg, Mn, Zn and Ni, and B is occupied by a trivalent cation such as Al, Cr, and Fe, are promising materials due to their wide range of applications including sensors for toxic gases, photocatalysts, magnetic materials, hard materials, and catalysts for the combustion of hydrocarbons [1-5]. Among spinel oxides, zinc chromite $ZnCr_2O_4$ has attracted attention for sensing and photocatalytic application, due to its excellent physical and chemical properties. There are several reports on the synthesis of $ZnCr_2O_4$ nanostructures, such as hydrothermal, mechanical, high-temperature solid-state reaction, and sol-gel methods [1-2]. Here, we present a synthesis of $ZnCr_2O_4$ nanofibers by an electrospinning process, characterized using scanning electron microscopy (SEM) and X-ray diffraction (XRD), with potential applications as biosensors.

In the synthesis, $Zn(NO_3)_2{\cdot}6H_2O$ and $Cr(NO_3)_3{\cdot}9H_2O$ in a 1:1 molar ratio were added to 20 mL of 50/50 (by volume) ethanol and N, N-dimethylformamide (DMF) mixture. The electrospinnable sol-gel was prepared by adding 2.0 g of polyvinylpyrrolidone (PVP) to the above solution and magnetically stirred overnight at room temperature. Then, the sol-gel solution was electrospun at an applied voltage of 18 kV with a flow rate of 0.5 mL/h, and the distance to collector was 17 cm. The collected electrospun samples were calcinated at 700 °C for 7 h at a 2 °C/min heating rate.

The morphology of the synthesized nanofiber was analyzed by SEM (Fig. 1a–c). The electrospun sample was composed of continuous and randomly oriented nanofibers (Fig. 1a), with a diameter in the range of 150–220 nm. After calcination, SEM images at a low and high magnifications are shown in Figure 1b and c, respectively. The 1-D morphology of particles were retained even after the calcination for 7 h; however, the diameter of nanofibers was reduced to 50–100 nm. Figure 1d demonstrated the X-ray diffraction (XRD) pattern of the final product, and all peaks were matched with JCPDS standard data file No. 00-022-1107, corresponding to $ZnCr_2O_4$. No secondary phase or impurities were detected by XRD. Thus, prepared nanofibers may have potential applications as biosensors and/or photocatalysts [6-8].

As a preliminary experiment, we tested the sensitivity of nanofibers for glucose electrochemically using CHI 760E electrochemical workstation. We observed a change in amperometric current at every addition of glucose as compared to bare carbon cloth (Fig. 2). The three-electrode system was used in an electrochemical experiment, where nanofiber-modified carbon cloth was used as a working electrode, platinum wire as the counter electrode, and Hg/HgO as reference electrode. The sensitivity will be optimized towards biomolecule sensors [9].

References

[1] Z. Mousavi, et al., Sci. Rep. **6** (2016), 20071. DOI: 10.1038/srep20071
[2] J.H. Kim, et al., Sens. Actuat. **B11** (2016), 498-506. DOI: 10.1016/j.snb.2016.05.104

[3] V.H. Nguyen, et al., Sep. Purif. Technol., (2021), 118511 (in press). DOI: 10.1016/j.seppur.2021.118511
[4] S. Chen, et al., J. Phys. Chem. **C117** (2013), 25019-25025. DOI: 10.1021/jp404984y
[5] H. Song, et al., ACS Catal. **11** (2017), 7610-7622. DOI: 10.1021/acscatal.7b01822
[6] K.D. Pelucarte, et al., Mater. Adv. (2022), in press. DOI: 10.1039/D1MA00984B
[7] S.R. Ede, et al., *ACS Catal.* **11** (2021), 4327-4337. DOI: 10.1021/acscatal.1c00465
[8] G. George, et el., J. Mater. Chem. **C7** (2019), 14949-14961. DOI: 10.1039/C9TC05068J
[9] This work was funded by NSF Excellence in Research (EIR) grant ECCS 1900837.

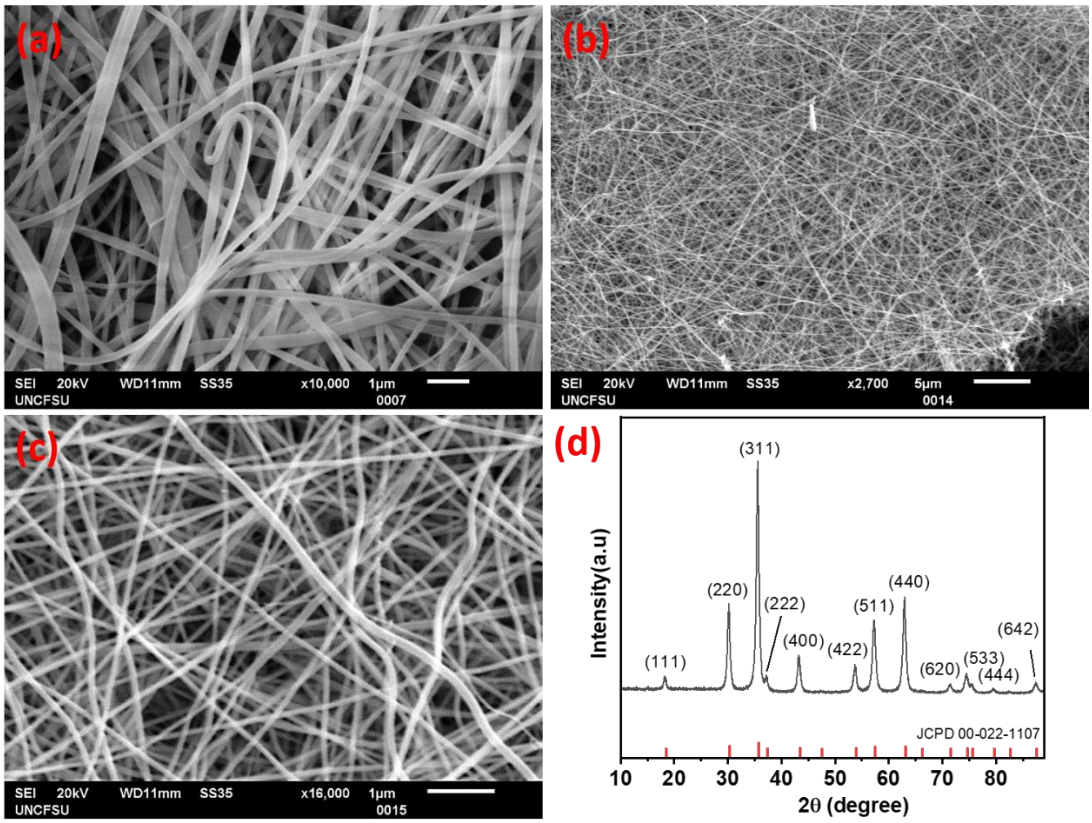

Figure 1. SEM image of $ZnCr_2O_4$ nanofibers before calcination (a) and after calcination (b, c); and XRD pattern of the calcinated nanofibers (d).

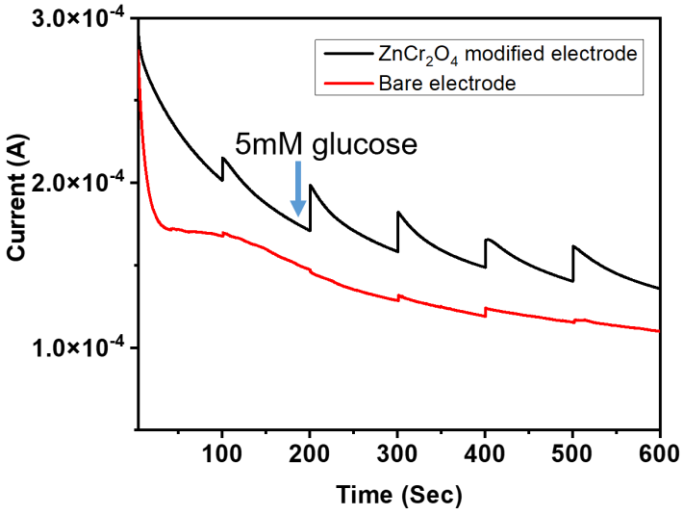

Figure 2. Amperometric *i-t* curve for glucose sensing using $ZnCr_2O_4$ nanofibers.

https://doi.org/10.1017/S1431927622010066 Published online by Cambridge University Press