ASEC 2025 Conference

The 6th International Electronic Conference on Applied Sciences

09–11 December 2025 | Online

MDPI

One-pot hydrothermal synthesis of Fe-doped ZnO nanoparticles

Diego-Antonio Corona-Martinez [1], C.M. Lopez-Badillo [1] *

[1] Facultad de Ciencias Químicas, Universidad Autónoma de Coahuila, Blvd. V. Carranza y José Cárdenas Valdés, C.P. 25280, Saltillo, Coah. México; Correspondence: cllopezb@uadec.edu.mx

INTRODUCTION & AIM

This study focuses on the production of Fe-doped zinc nanoparticles via one-pot hydrothermal synthesis, to achieve better control of crystallized nanoparticles via a single step without requiring any further purification or refinement of the crystalline structures.

To enhance the properties of the ZnO nanoparticles, doping of the wurtzite structure with Fe $^{3+}$ ions in quantities from 1 to 10% was used.

METHOD

The synthesis of ZnO nanoparticles under hydrothermal conditions was carried out to investigate the effects of the Fe^{3+} ion saturation on the production of ZnO nanoparticles and Fe-doped ZnO nanoparticles as a single phase without the formation of a secondary phase as the flowchart on Figure 1 indicates.

Therefore, the following analytical-grade precursor chemicals (Sigma Aldrich) were used: zinc nitrate hexahydrate ($Zn(NO_3)_2 \cdot 6H_2O$, CAS: 10196-18-6), sodium hydroxide (NaOH, CAS: 1310-73-2), and iron(III) chloride hexahydrate ($FeCl_3 \cdot 6H_2O$, CAS: 10025-77-1).

Before hydrothermal treatment, the stoichiometric amount of each powdered precursor reagent was weighed to produce ZnO nanoparticles.
In the case of Fe-doped ZnO nanoparticles, only the amount necessary to replace between 1% and 10% of the required zinc was used.

The powder mixture was placed in the PTFE container and dissolved with 30 mL of distilled water. Subsequently, the reagents were mixed for 30 minutes at 300 rpm using magnetic stirring.

The reactor vessel was sealed and subsequently heated in a conventional convection oven to 160 °C for various reaction intervals of 1 to 12 hours.
After treatment, the resulting reaction products were separated gravimetrically and vigorously washed four times with hot water at 60 °C.

The resulting powders were dried overnight at 80 °C and characterized using various techniques.

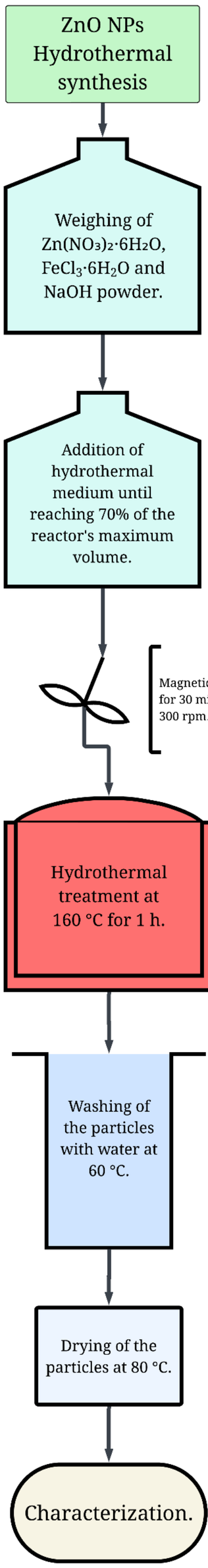

Figure 1.-Fe doped ZnO NPs synthesis process flowchart

REFERENCES

https://doi.org/10.1039/d3ma00053b
https://doi.org/10.21467/anr.3.1.28-39
https://doi.org/10.17509/ijost.v6ix
https://doi.org/10.1021/acsomega.1c03204
https://doi.org/10.1016/j.chphi.2023.100270
https://doi.org/10.1007/s10948-017-3992-x

RESULTS & DISCUSSION

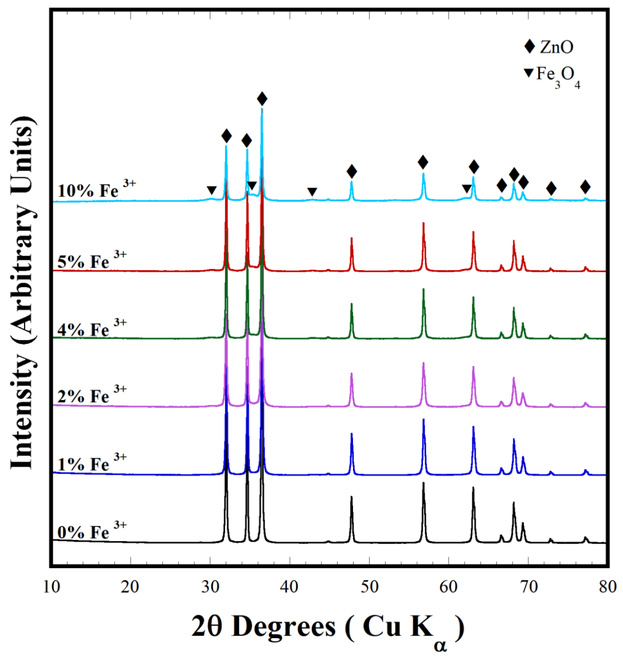

Figure 2 XRD patterns of Fe doped ZnO NPs prepared for 1 h at 160 °C increasing Fe^{3+}doping.

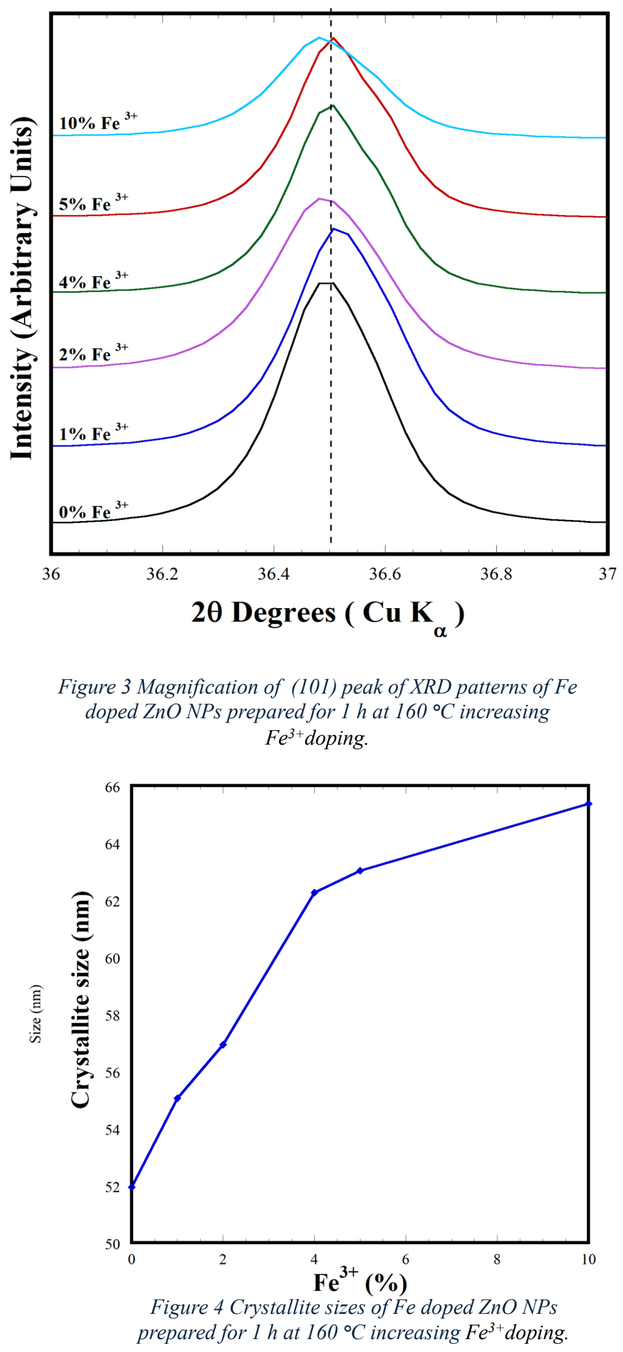

Figure 3 Magnification of (101) peak of XRD patterns of Fe doped ZnO NPs prepared for 1 h at 160 °C increasing Fe^{3+}doping.

Figure 4 Crystallite sizes of Fe doped ZnO NPs prepared for 1 h at 160 °C increasing Fe^{3+}doping.

The X-ray diffraction analysis shown in figure 2 clearly indicates the formation of ZnO particles, since all the peaks match the standard pattern for ICSD card 01-089-7102 (Armah et al., 2020) corresponding the ZnO in the hexagonal (wurtzite) structure. No other relevant peaks are observed, which means almost no secondary phases on the precipitation of the powders.

However, on the 10% Fe^{3+} doped sample there are slight changes in the base line of the XRD pattern corresponding to the Fe_3O_4 phase (JCPDS 96-900-6190) (Garskaite et al., 2021). Nevertheless, they are not high and defined enough to be considered a secondary phase, but the limit of solubility of the Fe^{3+} ions in the system, like what is shown on the work of (Rao et al., 2023).

Although all the identified peaks are high in counts and sharp in shape, indicating high crystallinity of the particles(Sekhar et al., 2017) and good crystallinity of doping. Another interesting effect worth of mentioning shown in the magnification of the XRD patterns, (Figure 3), is the small broadening trend of the peaks as the Fe content increases, suggesting an enhancement of the disorder of the ZnO matrix consistent with the work of Aragón et al., 2023.

The broadness of the base of the peaks still small this indicate a small crystallite size, since the Fe content increases in the system the resultant particles growth from 52 to 65 nm, these parameters are presented in Figure 4 and were obtained from Debye-Scherer´s formula (Fatimah et al., 2021).

CONCLUSION

This works shows the successful one-pot hydrothermal synthesis of Fe-doped ZnO NPs presenting the wurtzite structure. The XRD measurements reveals the effect of the Fe content on the crystalline structure showing a decrease of crystallinity and increase of the grain size without the formation of secondary phases on the hydrothermal media, making this synthesis route a promising pathway to produce doped materials.