

Facile hydrothermal synthesis of $Zn_{1-x}Fe_xO$ nanoparticles

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INTRODUCTION & AIM

The aim of this research was to obtain a facile methodology to generate nanoparticles of $Zn_{1-x}Fe_xO$ solid solution NPs, with the inclusion of Fe^{2+} ions in various quantities on the zinc oxide structure by hydrothermal synthesis in mild conditions.

METHOD

Synthesis of Fe doped ZnO nanoparticles

Introduce $Zn(NO_3)_2 \cdot 6H_2O$, $FeCl_2 \cdot 4H_2O$ and NaOH powders in the reactor.

Add the hydrothermal media to reach 70% of the reactor volume.

Homogenize by magnetic stirring for 30 minutes at 300 rpm.

Heat the reactor at 160 °C for 1 h.

Remove the hydrothermal media.

Dry the nanoparticles.

Characterize the powders.

The synthesis of $Zn_{1-x}Fe_xO$ nanoparticles was carried out under hydrothermal conditions to determinate the viability to introduce Fe^{2+} ions on the 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(II) chloride hexahydrate ($FeCl_2 \cdot 4H_2O$, CAS: 13478-10-9).

Before the hydrothermal treatment, a stoichiometric amount of each powdered precursor reagent was weighed to produce ZnO nanoparticles.

For the Fe-doped ZnO nanoparticles, only the amount necessary to replace between 0% and 10% of the required zinc was used.

The powder mixture was placed in the PPL container and dissolved with 30 mL of tri distilled water.

Subsequently, the reagents were homogenized for 30 minutes at 300 rpm using magnetic stirring.

Then the reactor vessel was sealed and subsequently heated in a conventional convection oven to 160 °C for 1hour.

After treatment, the resulting reaction products were separated gravimetrically and vigorously washed four times with a mix of hot water and ethanol (70/30 ratio) at 60 °C.

The resulting powders were dried overnight at 80 °C and characterized using various techniques to confirm the formation of the $Zn_{1-x}Fe_xO$ solid solution NPs.

RESULTS & DISCUSSION

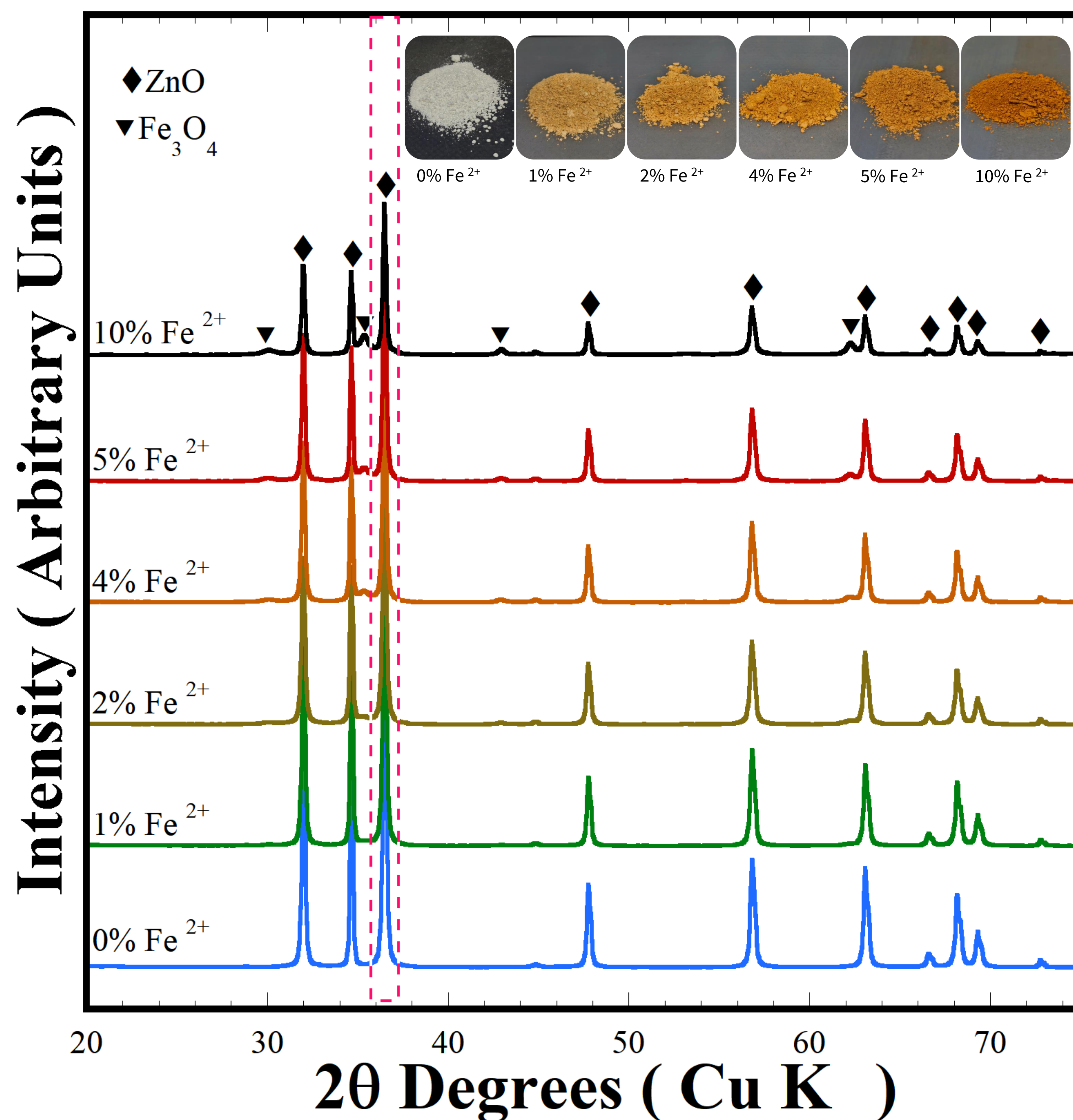


Figure 2 XRD patterns of Fe doped ZnO NPs prepared for 1 h at 160 °C increasing Fe^{2+} content.

The X-ray diffraction pattern 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 to the ZnO in the wurtzite structure.

Moreover, the doped NPs exhibited a change in color and hue relative to the ZnO NPs from white to indochine (C97305 hex). Also, above 5% Fe^{2+} doped sample there are very small peaks matching the Fe_3O_4 phase (JCPDS 96-900-6190) (Simmons et al., 2015), indicating the limit of solubility of the Fe^{2+} ions in solid solution

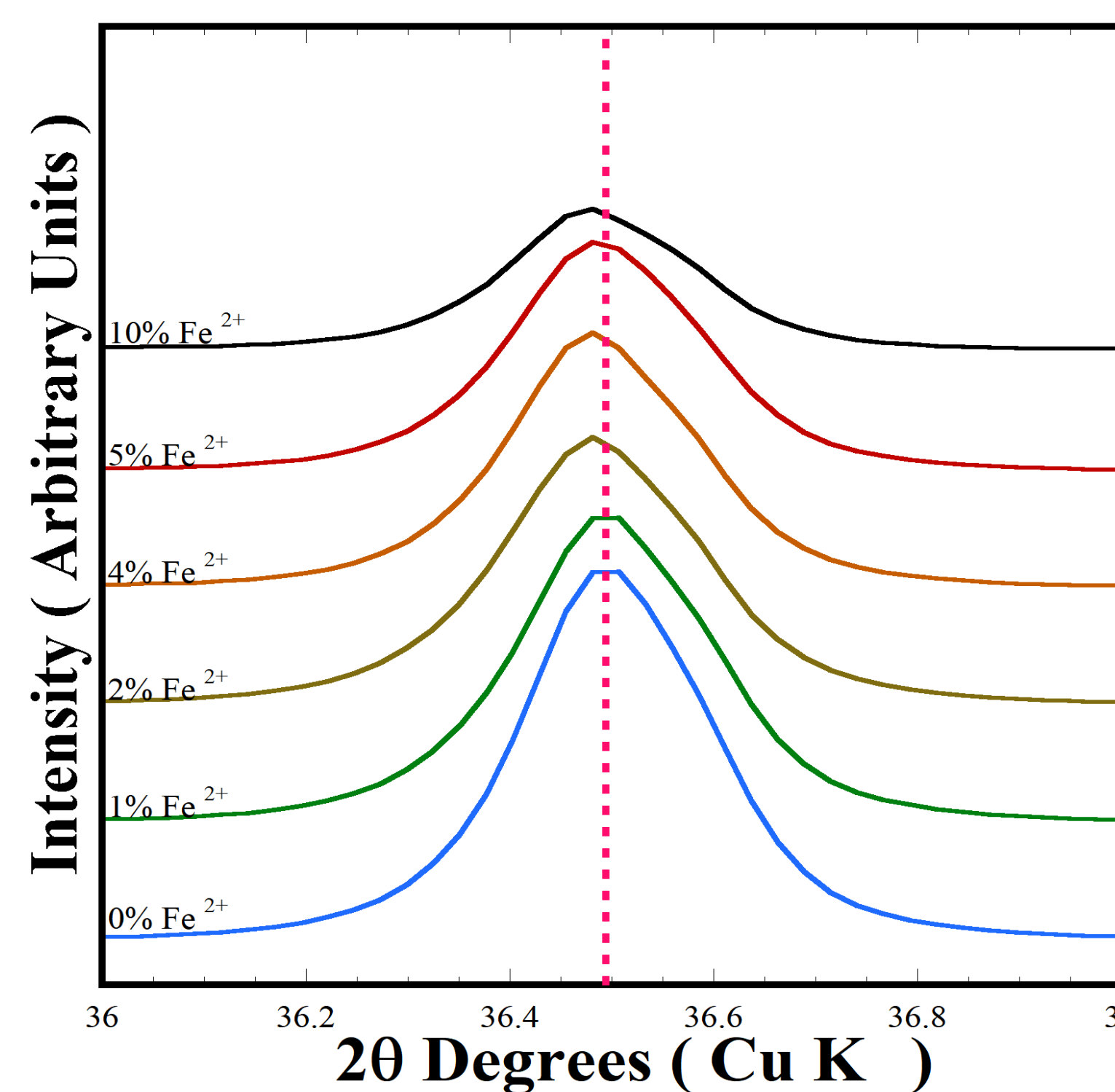


Figure 3 Magnification of (101) peak of XRD patterns of Fe^{2+} doped ZnO NPs

Furthermore the magnification of the main peak (Figure 3), shown the broadening and shortening of the peaks as the Fe^{2+} content increases, suggesting an increment of the disorder of the ZnO crystalline matrix and their gradual shift to lower angles, indicates an expansion of the lattice of the unit cell, (Imboon et al., 2025) consistent for the obtained from Debye-Scherrer's formula where the particles go from 45 to 53 nm with 10% Fe^{2+} substitution.

REFERENCES

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CONCLUSIONS

This investigation showed the success of the proposed methodology to fabricate in a consistent and efficient way nanoparticles of $Zn_{1-x}Fe_xO$ solid solution, opening the possibility to fine tune the Fe^{2+} content in the ZnO particles, without the precipitation of another crystalline phase. This can be leveraged in various applications, as well as the ease of carrying out the synthesis at low temperatures and in short times.

Figure 1.- $Zn_{1-x}Fe_xO$ solid solution NPs synthesis process flowchart.