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A comparative Raman spectroscopy and XRD assessment of lattice transitions and impurities in Mn and Zn doped ferrite nanoparticles

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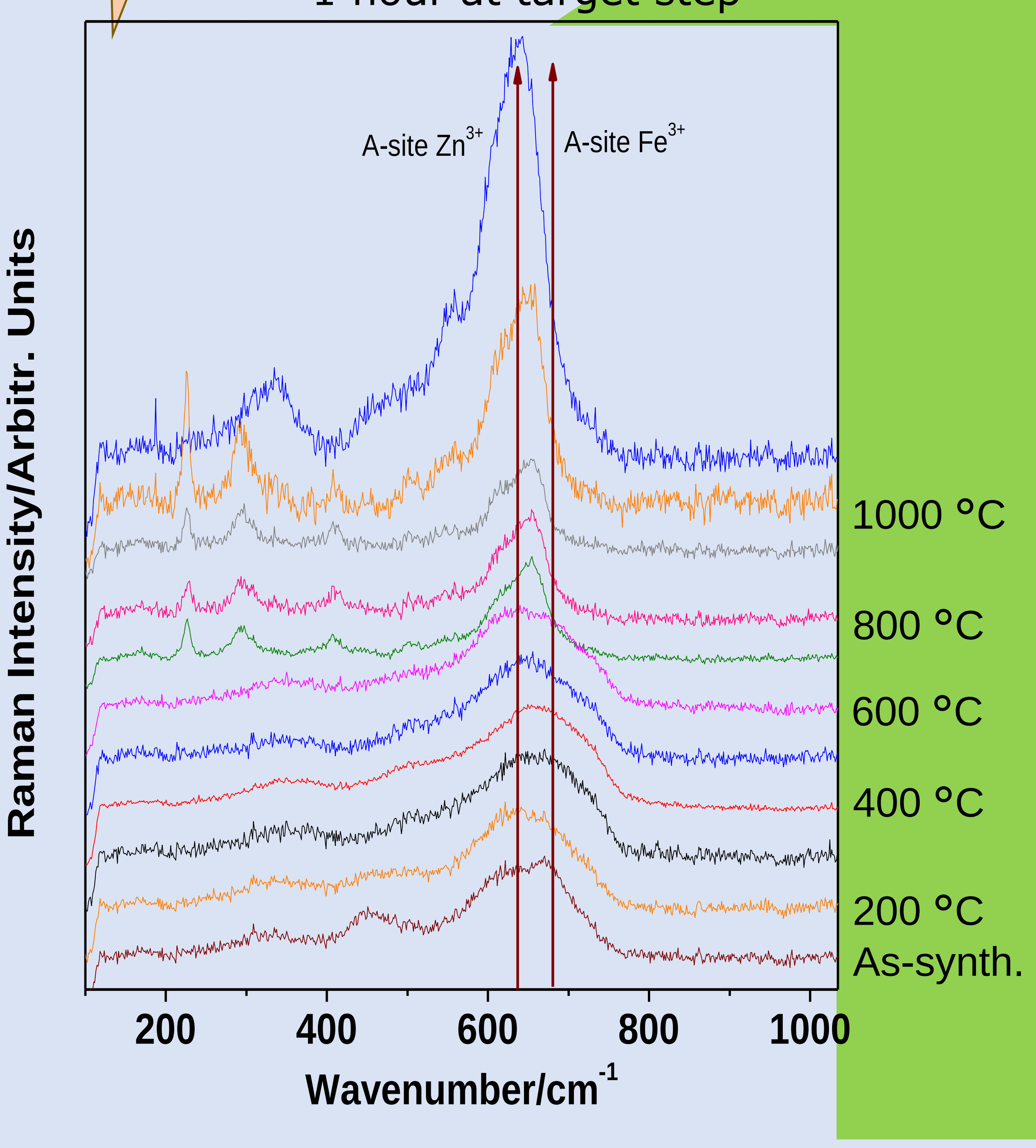
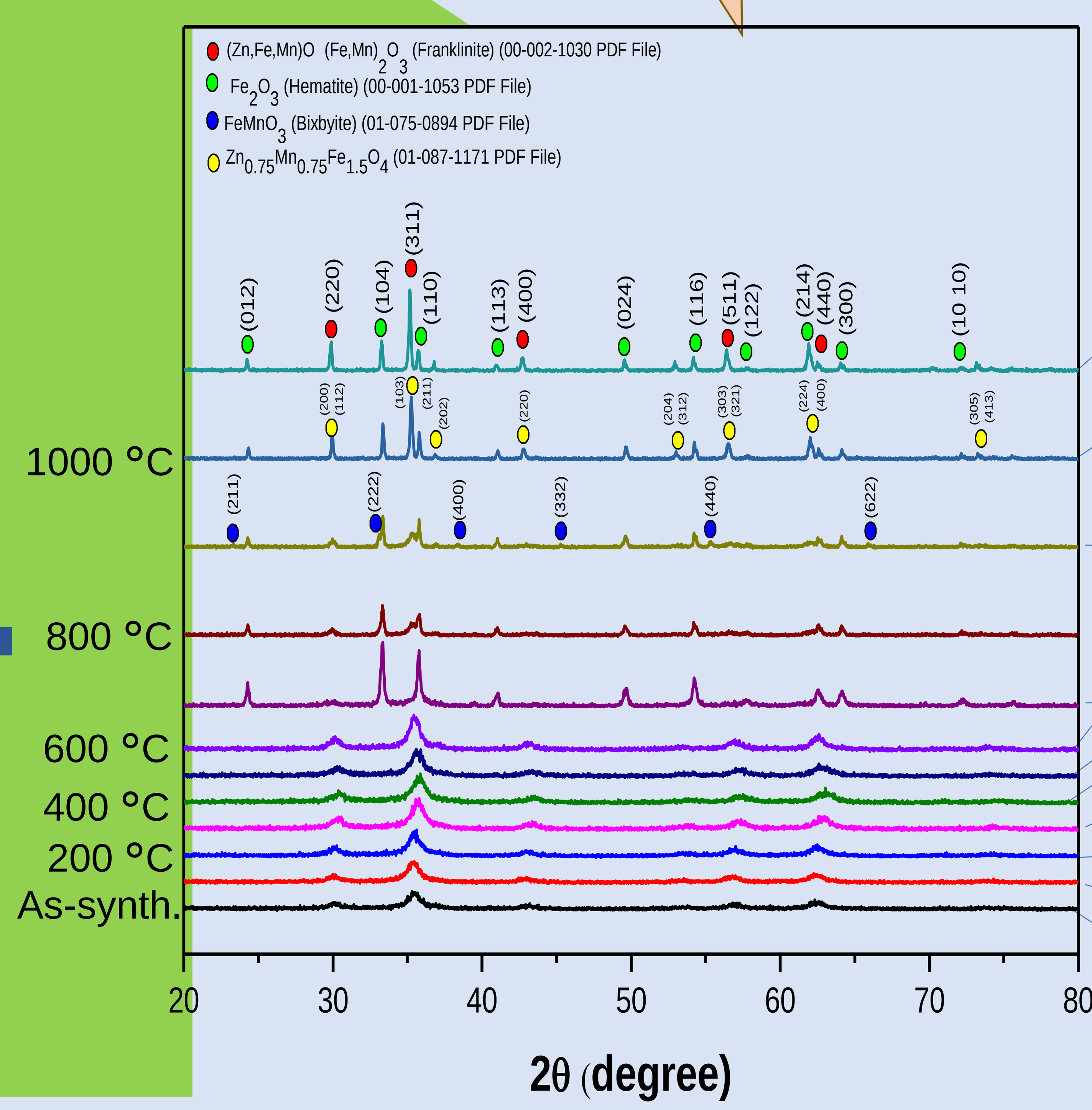
Raman spectra

$Mn_{0.8}Zn_{0.2}Fe_3O_4$ nanoparticles
100 to 1100 °C, 100 °C step,
1 hour at target step



Temperature

X-ray diffraction patterns



Major phase:
Mg–magnetite
Mh-maghemite
H-hematite

M_S (emu/g)

M_S (emu/g)	Major phase
31.7	Mg
39.6	Mg
none	H
43.8	Mh
40.2	Mh
42.3	Mh
44.2	Mh
50.3	Mg
49.5	Mg
48.5	Mg

SCOPE

Temperature-induced phase transitions and the presence of impurities to the spinel phase ferrite nanoparticles were comparatively assessed by Raman spectroscopy and X-Ray Diffraction, in order to point out advantages and disadvantages of using either method depending on the research issue at hand. Temperatures steps considered separately: as-synthesized, 100, 200, 300, 400, 500, 600, 700, 800, 900, 1000 and 1100 °C.

Raman spectroscopy revealed gradual phase transitions from predominantly magnetite-like below 300 °C, over maghemite-like in 300-600 °C, hematite-dominated in the 700-1000 °C range, and back to a spinel phase at 1100 °C with different arrangement of cations on A-sites than the starting sample (see graph)

X-ray diffraction on the same samples showed abrupt transition from the spinel structure to rhombohedral (hematite) between 600 and 700 °C, and back to spinel at 1100 °C, but this technique performed better at revealing oxide phases present below the identification threshold of Raman spectroscopy.

Magnetization measurements support the phase transitions, showing no saturation magnetization for the samples annealed at 700 – 900 °C, while those annealed at 1000 and 1100 °C re-gained saturation magnetization up to 31.7 emu/g.

Magnetite- and maghemite-like structures in the 100 to 300 °C range are more clearly distinguished by Raman than XRD, due to their isostructural character (both have face-centered cubic symmetry).

CONCLUSION

From the analytical perspective, it is important to know what details should be evaluated and which methods are the most suitable for those kinds of investigations. In this paper we have shown the specific advantages and drawbacks of Raman spectroscopy and X-ray diffraction applied to the same $Mn_{0.8}Zn_{0.2}Fe_2O_4$ nanoparticle samples. Raman proved to perform excellently in detecting cation vacancies and subsequently differentiating between isostructural magnetite and maghemite, while XRD performs better in trace-amount phase recording.

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References contained in manuscript in preparation, *A comparative Raman spectroscopy and XRD assessment of lattice transitions and impurities in Mn and Zn doped ferrite nanoparticles;* authors Nekvapil, F., Bunge, A., Bortnic, R.A., Leoștean, C., Barbu-Tudoran, L., Turcu, R.