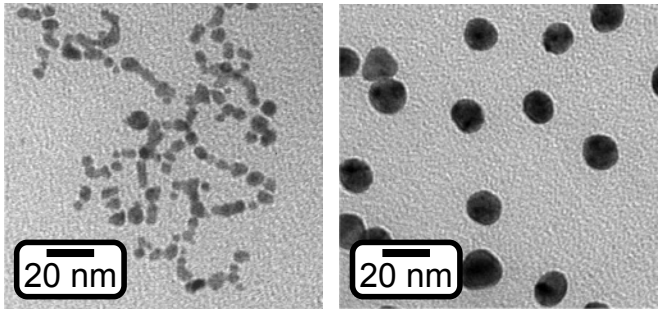
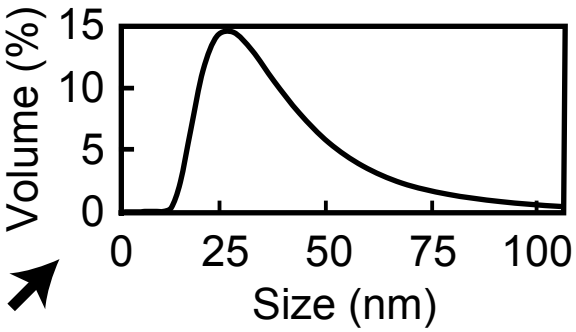
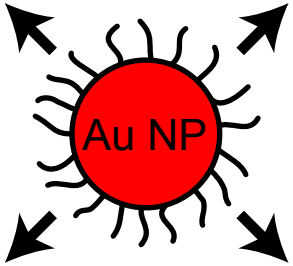
Au NP Characterization

TEM → size and morphology

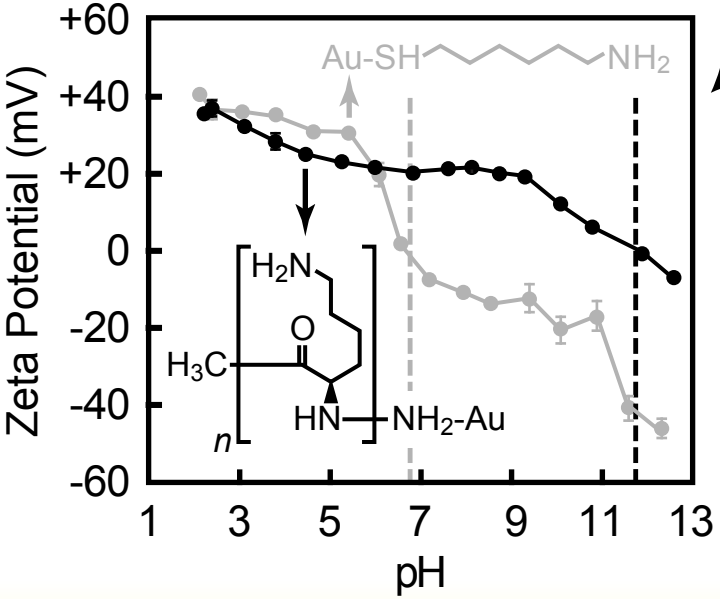


DLS → hydrodynamic diameter

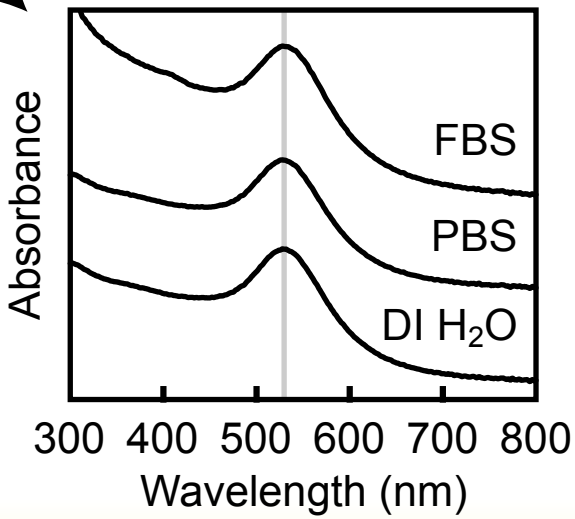




DLS → zeta potential



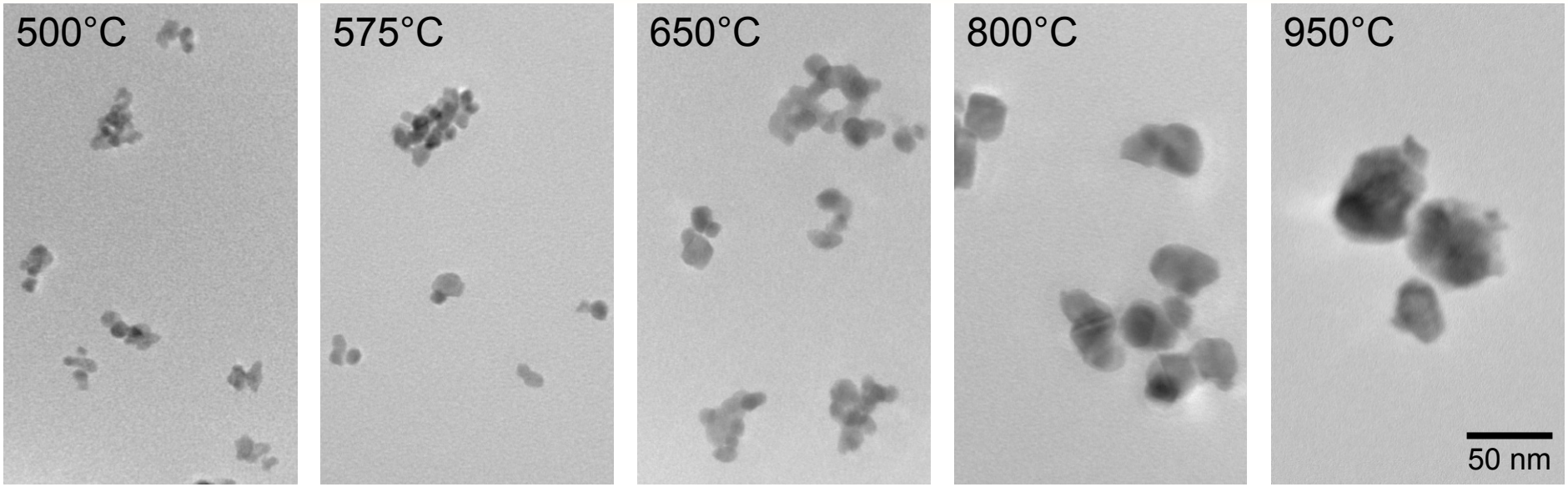
UV-vis → SPR peak



Cole et al., Nanomedicine, 2015

UNIVERSITY OF NOTRE DAME
http://www.nd.edu/~bioeng

TEM vs. XRD of HfO_2 Nanoparticle Size

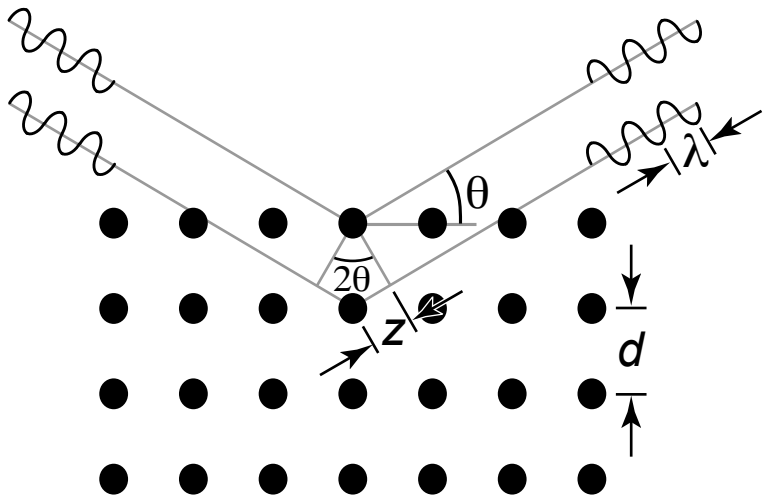


Temperature (°C)	XRD Crystallite Size (nm)	TEM Diameter (nm)	TEM Aspect Ratio
500	7.2 (0.8)[a]	7.4 (1.6)[a]	1.3 (0.2)[a,b]
575	8.4 (1.0)[a]	9.1 (2.3)[a]	1.3 (0.2)[a]
650	12.3 (1.0)[b]	12.5 (3.2)[b]	1.2 (0.2)[b]
800	21.2 (2.0)[c]	22.5 (5.6)[c]	1.2 (0.1)[b]
950	32.8 (1.3)[d]	31.0 (8.2)[d]	1.2 (0.1)[a,b]

McGinnity *et al.*, *Nanoscale*, 2016

X-Ray Diffraction (XRD)

x-ray detector



x-ray source: Cu Kα radiation (λ = 1.54 Å)

- monochromatic
- parallel
- coherent (in phase)

constructive interference requires:

$n \cdot \lambda = 2z$ where $z = d \cdot \sin\theta$

Bragg's Law: $n \cdot \lambda = 2d \cdot \sin\theta$

where d = interplanar spacing

$$d = \frac{a}{\sqrt{h^2 + k^2 + l^2}} \quad \text{(cubic)}$$

a = lattice parameter

hkl = Miller indices

used predominately in inorganic crystalline materials to determine:

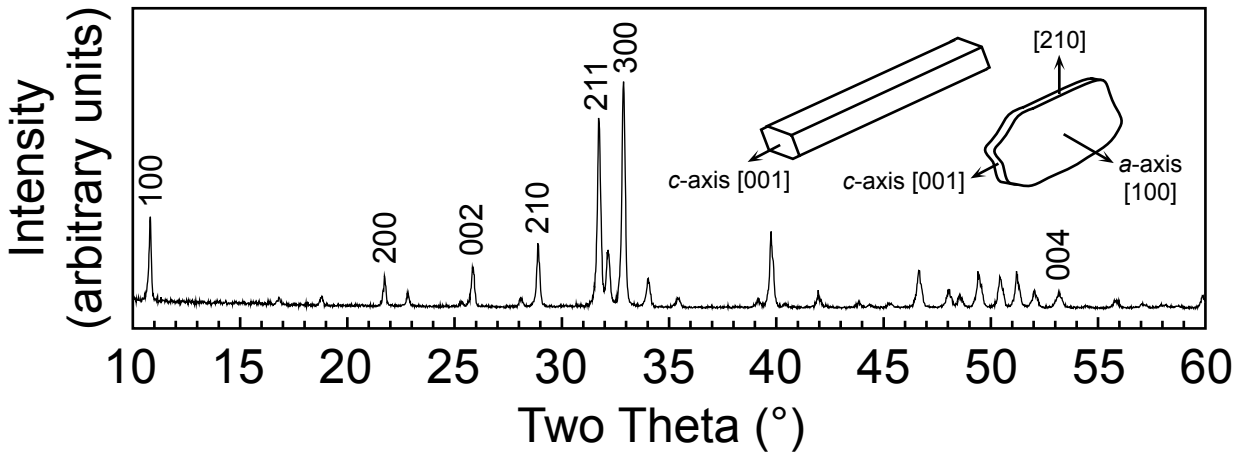
- phase identification (crystal structure)
- composition (phase fractions)
- crystal size → $t = \dfrac{0.9\lambda}{B \cdot \sin\theta}$ where $B^2 = B_m^2 - B_s^2$
- lattice parameters
- crystal orientation (texture analysis)
- residual stresses

B_m = FWHM peak breadth

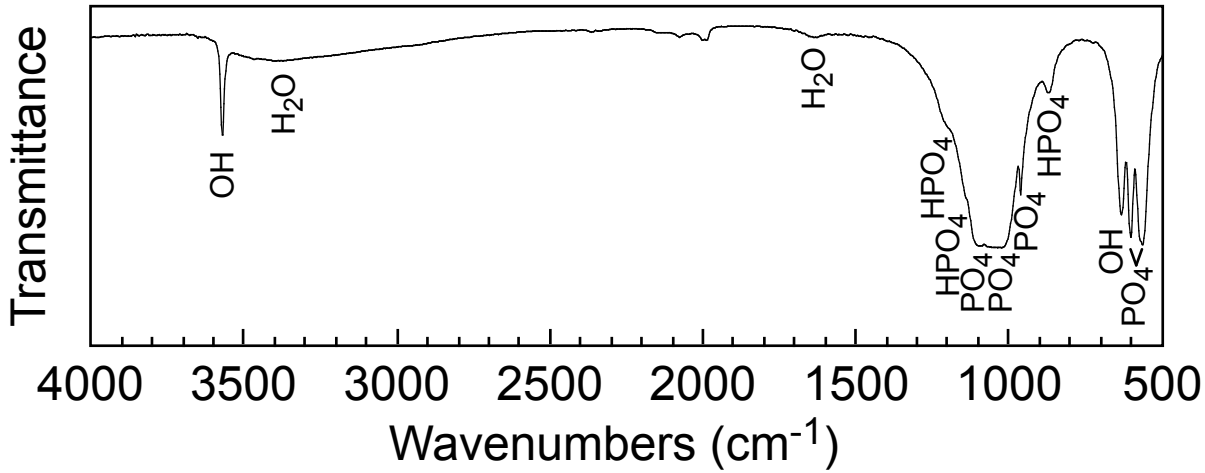
B_s = that of a ref. material with crystal size > 0.1 μm

XRD & FTIR Spectra for Hydroxyapatite

XRD

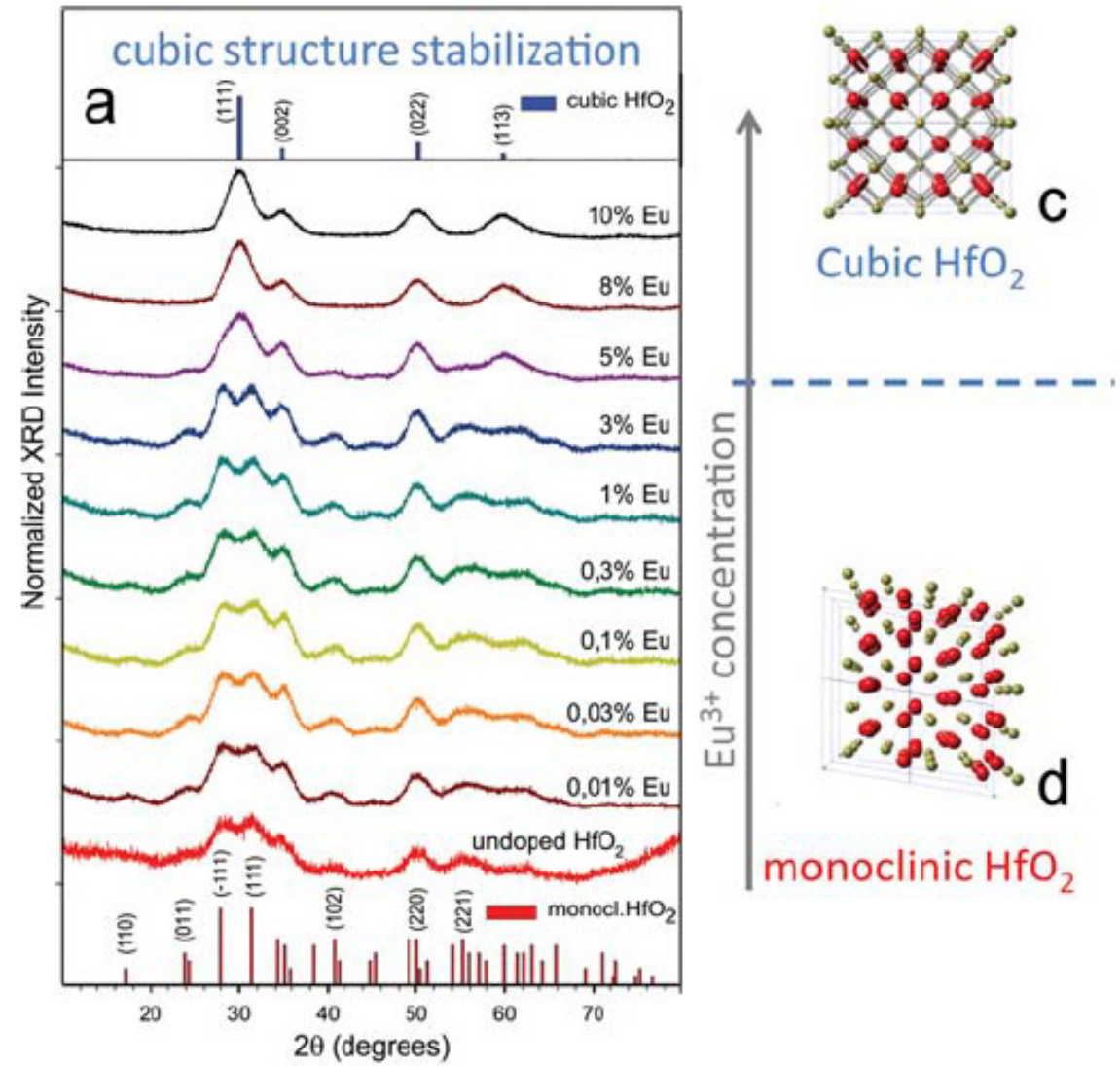


FTIR



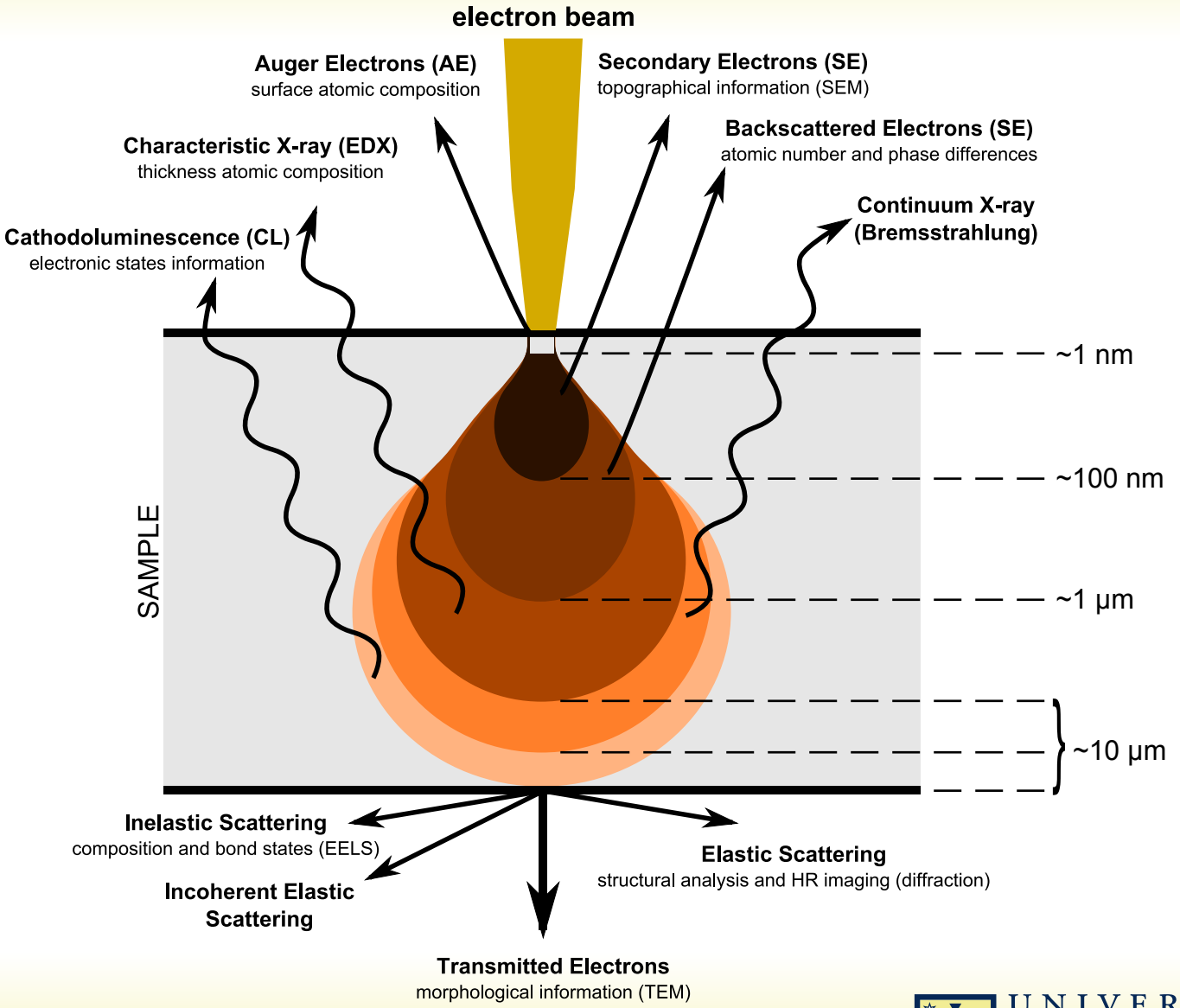
Roeder *et al.*, *J. Am. Ceram. Soc.*, 2006

EM & XRD Characterization Eu-HfO_2 NPs



Lauria *et al.*, *ACS Nano*, 2013

Interaction of Electron Beam with Materials

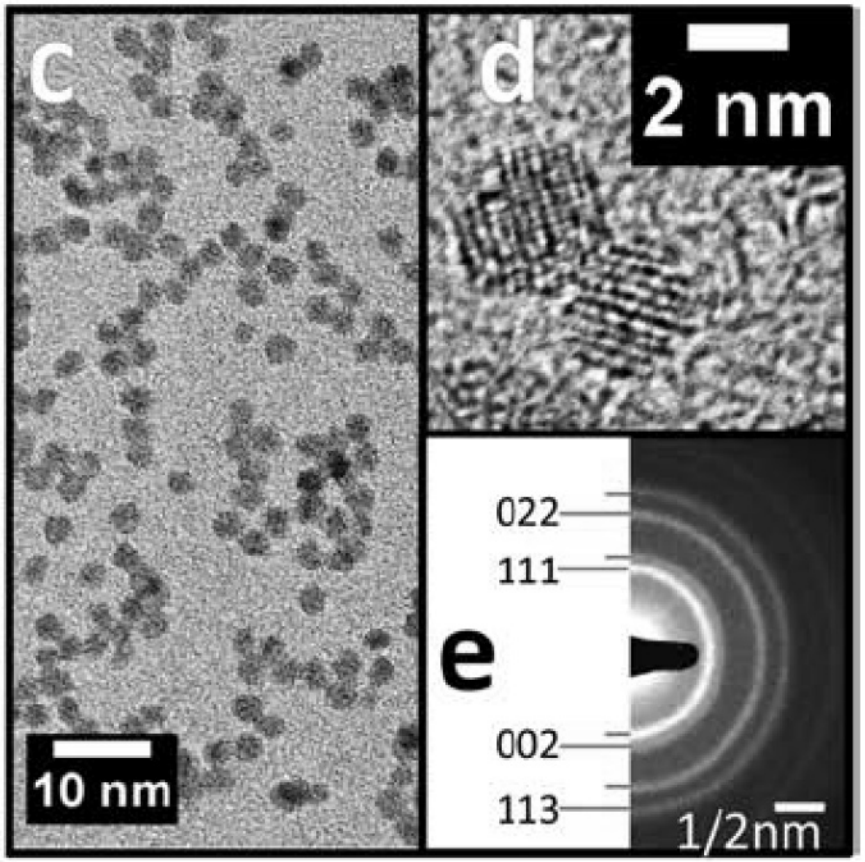


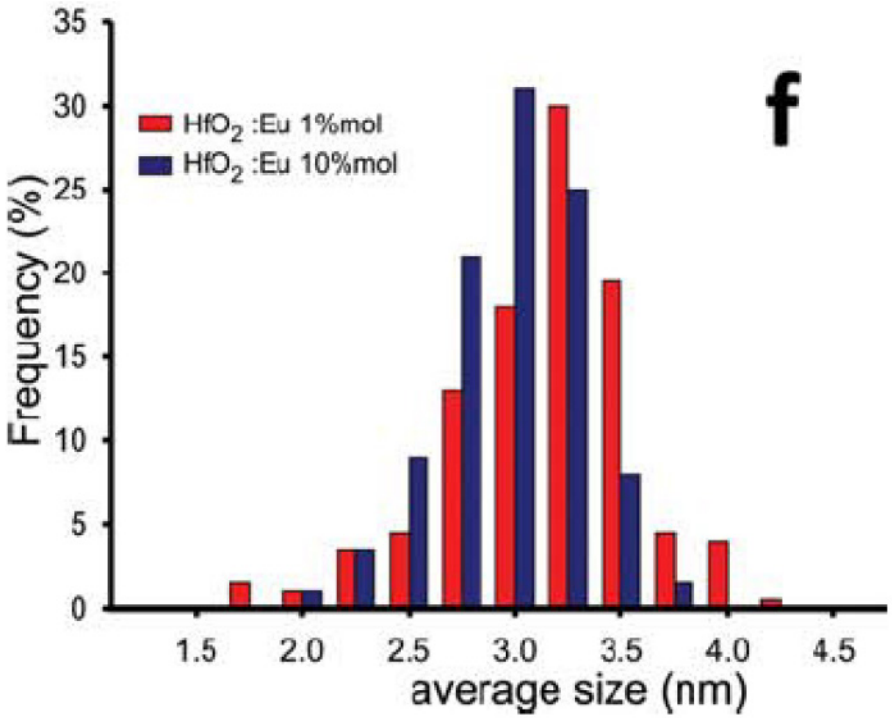
Adapted from Creative Commons, 2013

UNIVERSITY OF NOTRE DAME

http://www.nd.edu/~bioeng

EM & XRD Characterization Eu-HfO_2 NPs

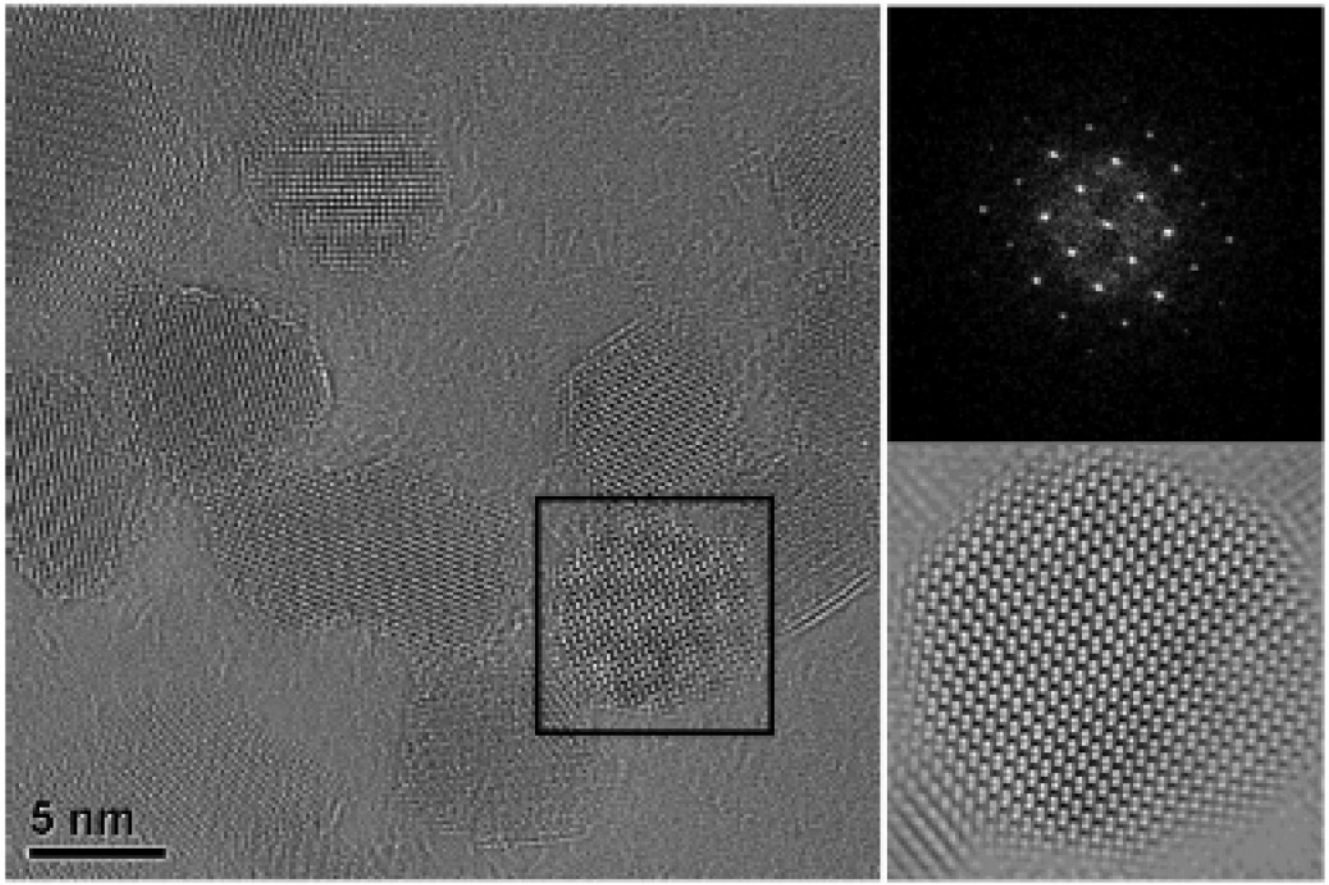




Lauria *et al.*, *ACS Nano*, 2013

EM Characterization of $Co_{0.94}Fe_{3.30}O_4$ NPs

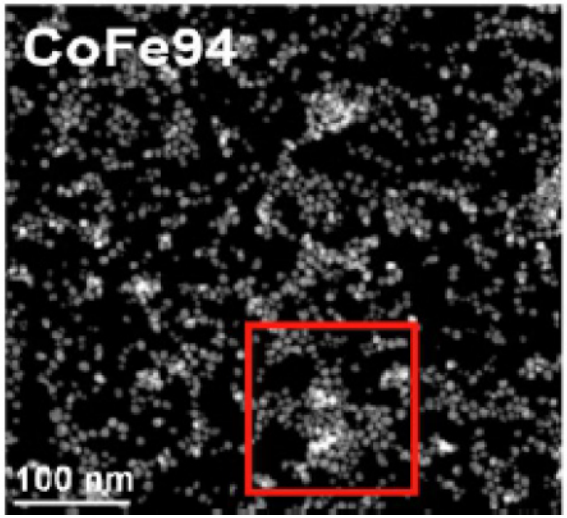
HRTEM

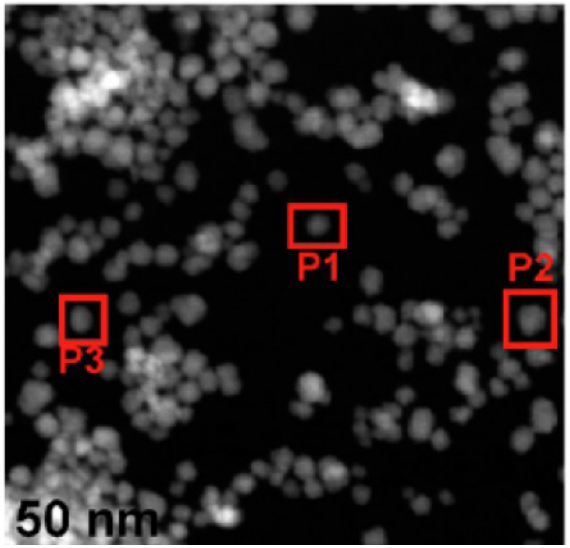


Carta *et al.*, *J. Phys. Chem. C.*, 2013

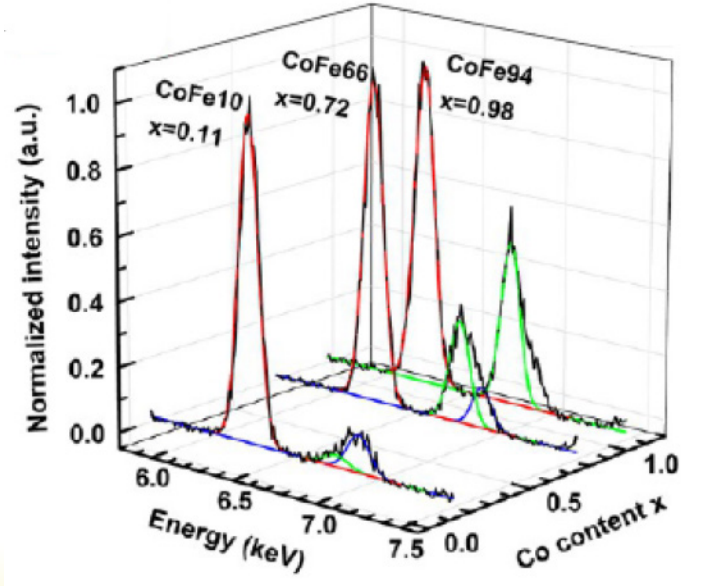
EM Characterization of $Co_{0.94}Fe_{3.30}O_4$ NPs

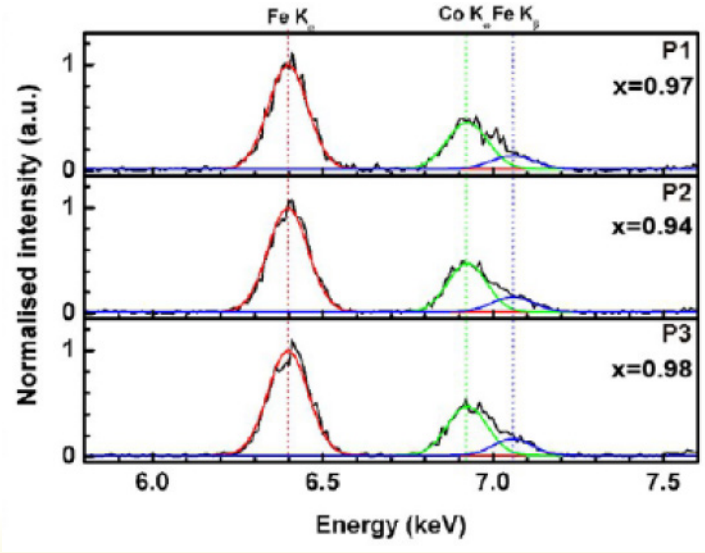
STEM





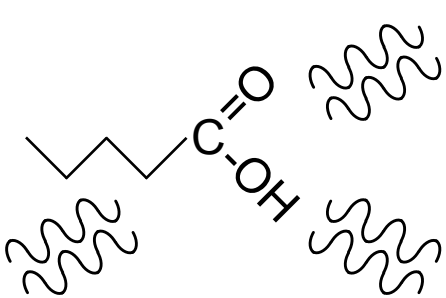
EDS





Carta *et al.*, *J. Phys. Chem. C.*, 2013

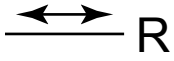
Fourier Transform Infra-Red Spectroscopy (FTIR)

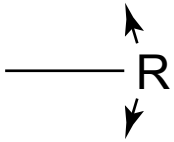


infra-red radiation of intensity, I_1

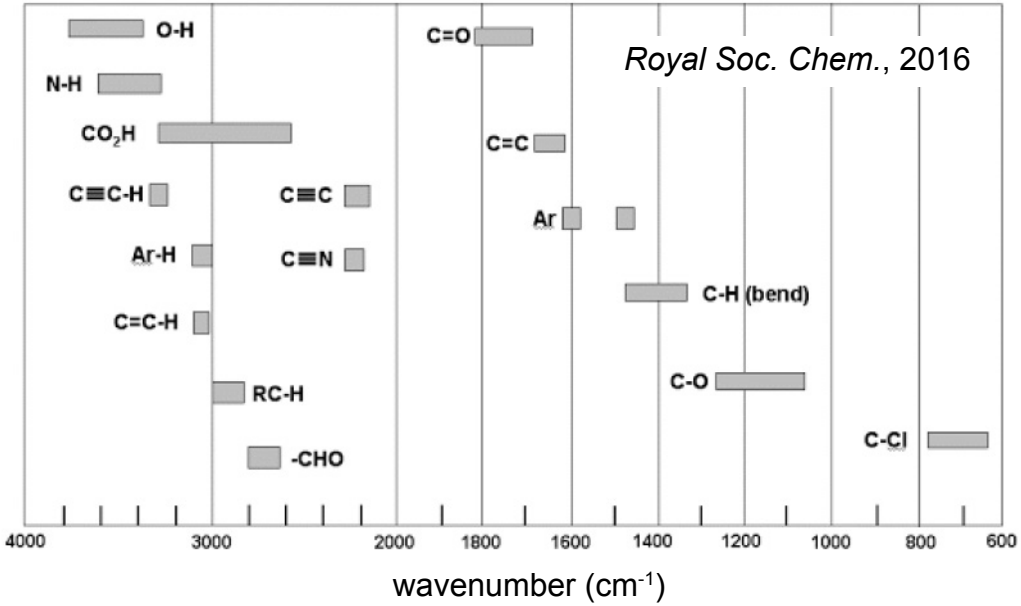
transmitted or reflected, I_2

energy absorbed by characteristic molecular vibrations

e.g., stretching 

bending 

etc.

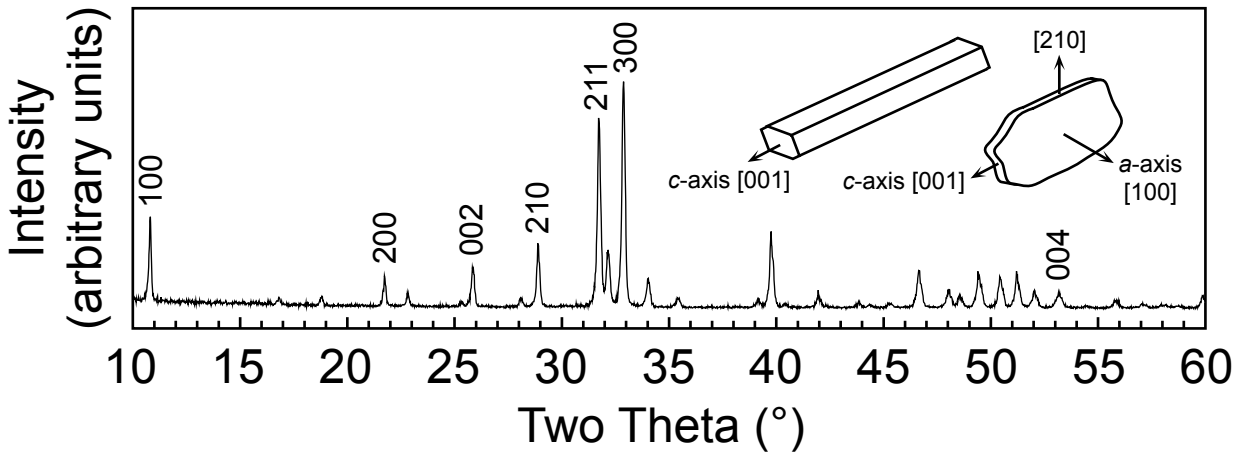


used predominately in organic molecules and macromolecules to determine:

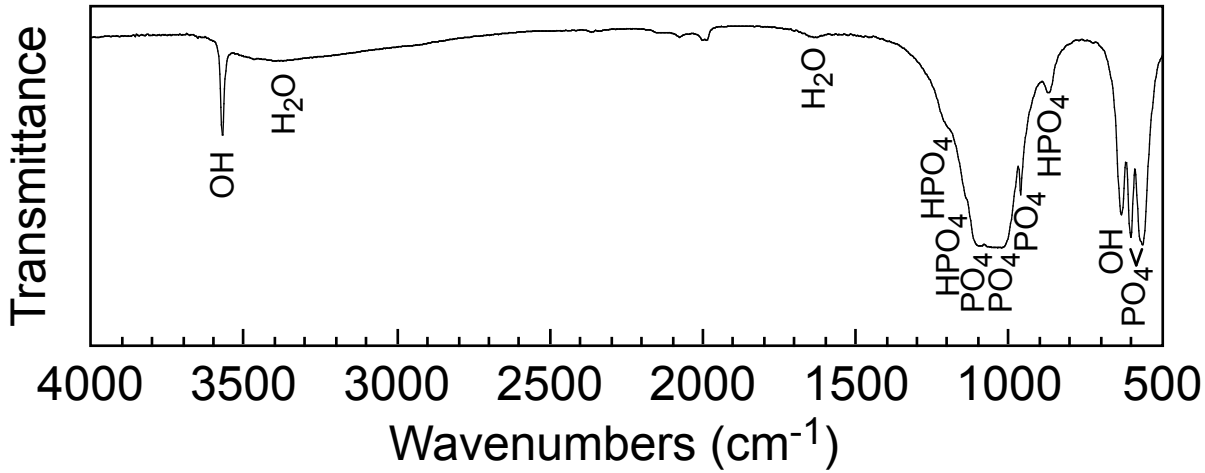
- ligand identification (molecular structure)
- composition

XRD & FTIR Spectra for Hydroxyapatite

XRD



FTIR



Roeder *et al.*, *J. Am. Ceram. Soc.*, 2006