

Microwave-Assisted Hydrothermal Synthesis of Ceria Nanorods: Reaction Temperature Study



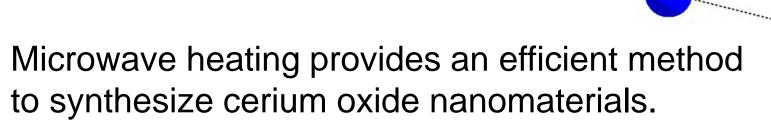
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Introduction

Cerium (IV) Oxide (CeO_{2-x}, $0 \le x \ge 0.5$) is an important industrial catalyst used in:

- Petroleum Refining
- Catalytic Converters
- Organic Chemical Synthesis
- Fuel Cells
- Chemical Sensors

Defect sites have a large influence on the catalytic activity of cerium oxide.

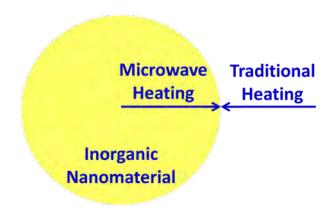


The effect of the unique heating profile provided by microwaves on the resulting nanostructures, particularly on the types and concentrations of defects, merits further investigation.

Aims

- 1. Investigate the effect of reaction temperature on the morphology of cerium oxide nanomaterials synthesized by the microwave hydrothermal method.
- 2. Characterize the defects present on the as-synthesized cerium oxide nanomaterials.

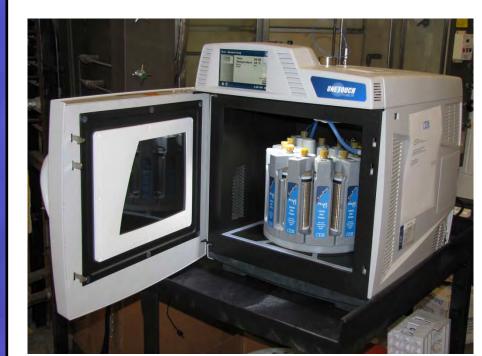
Method



The microwave hydrothermal process provides:

- Unique heating profiles
- Shorter synthesis times
- Variation in nanomorphology
- Reactants: Ce₂(SO₄)₃ and 10 M NaOH
- Temperature: 80 °C 200 °C
- Temperature Ramp Rate: 2 °C/min.
- Reaction Time: 1 hour
- Pressure: 0 psig 160 psig





Microwave Model: CEM Mars 6

Maximum Power: 1800 W

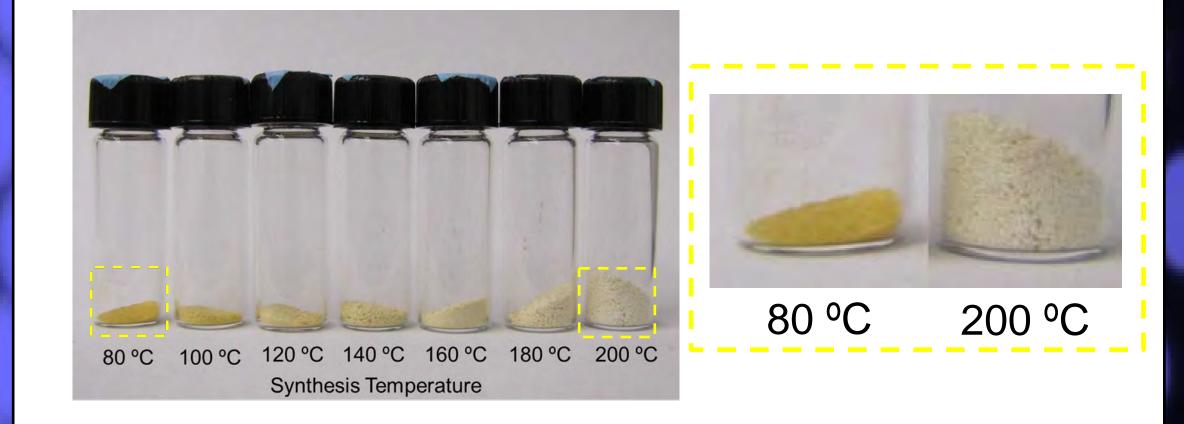
Reaction Vessel: CEM EasyPrep

Vessel Material: Teflon

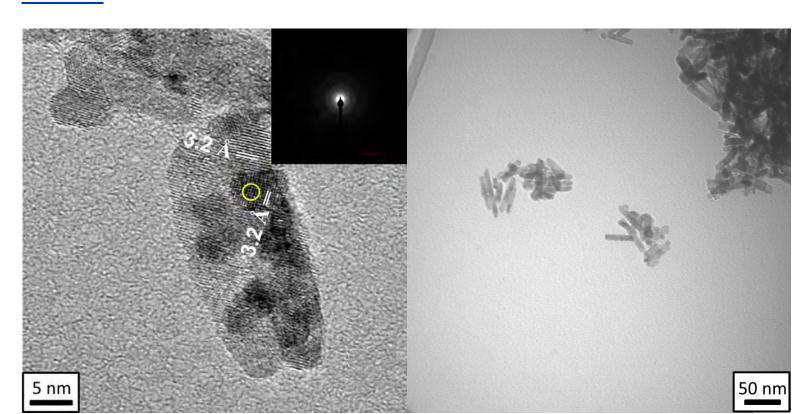
Vessel Volume: 100 mL

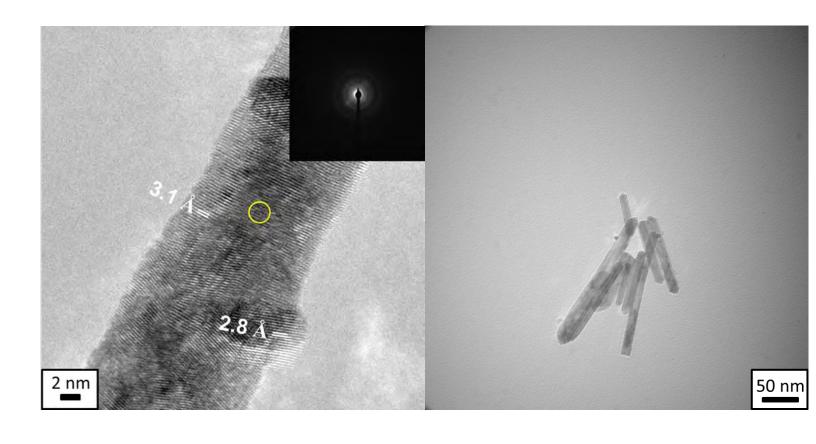
Results

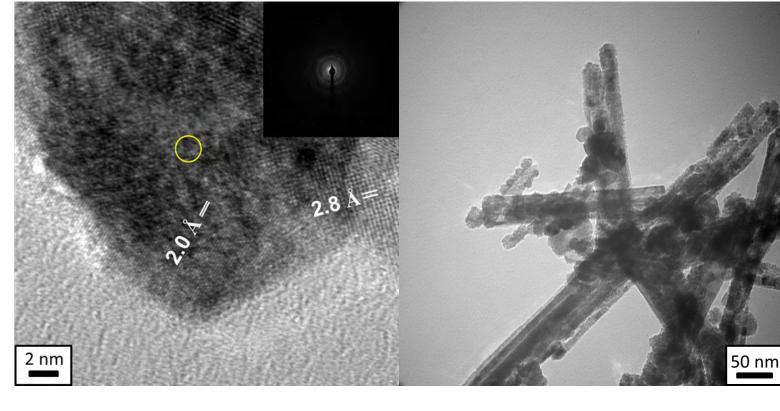
Sample Appearance



TEM







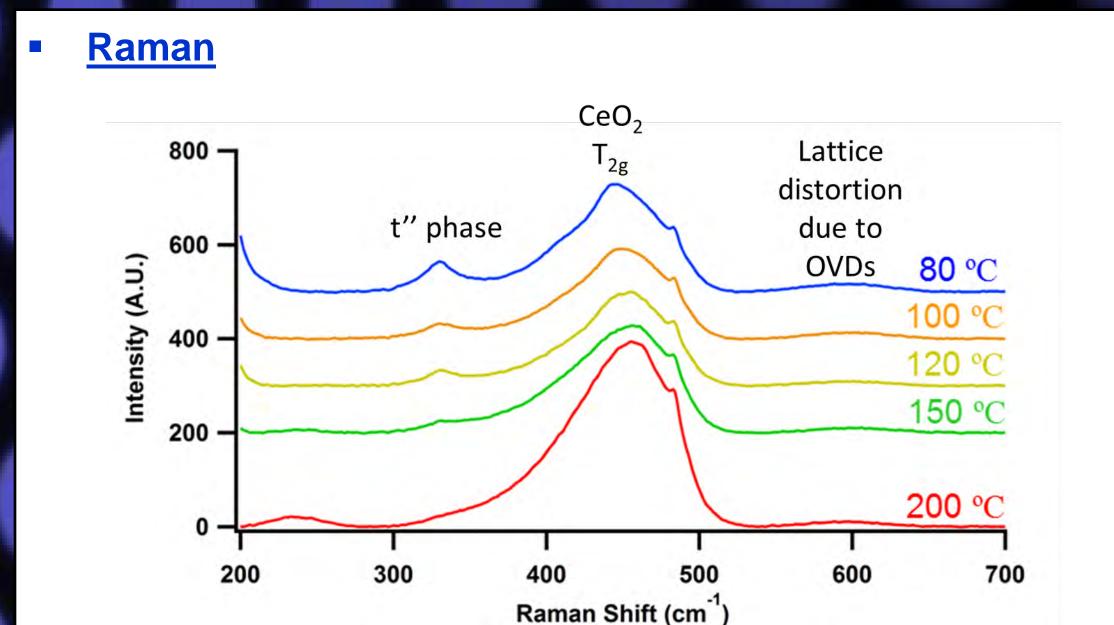
200°C

120°C

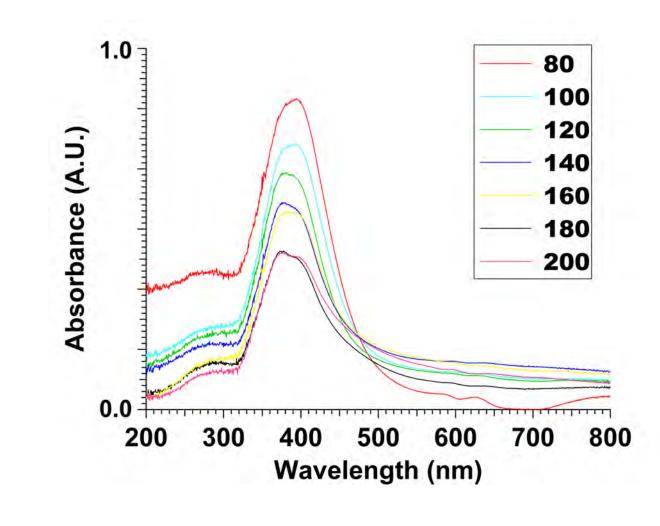
• XRD

Synthesis Temperature (°C)	Lattice Parameter (Å)	800 CeO ₂
80	5.42901 (+/00106)	(111)
100	5.43776 (+/00088)	(1) 600 − (1) (1) (1) (1) (1) (1) (1) (1) (1) (1)
120	5.43666 (+/00053)	CoO
140	5.43506 (+/00057)	$\frac{1}{2}$ $\frac{1}$
160	5.43826 (+/00035)	= 200 -
180	5.43974 (+/00032)	100 - Holy Market Marke
200	5.42276 (+/00035)	20 30 40 50
		Two Theta (degre

Lattice parameters in table found by Rietveld analysis using FullProf Suite.



<u>UV-Vis</u>



Conclusions

- The microwave hydrothermal method can be used to rapidly produce CeO_{2-x} nanorods of varying size.
- The color of the as-synthesized nanomaterials changes with temperature.
- Distortion of the cerium oxide lattice decreases with increasing temperature.

References

- 1. N.J. Lawrence, J.R. Brewer, L. Wang, T.S. Wu, J.M Wells-Kingsbusy, M.M Ihrig, G. Wang, Y.L. Soo, W.N. Mei, C.L. Cheung, *Nano Letters* **11**, 2666-2671 (2011).
- 2. M. Baghbanzadeh, L. Carbone, P.D. Cozzoli, C.O. Kappe, *Angewandte Chemie* **50**, 11312-11359 (2011).
- 3. F. Zhang, C. Chen, J.C. Hanson, R.D. Robinson, I.P. Herman, S. Chan, *J. Am. Ceram. Soc.* **89**, 1028-1036 (2006).

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