

## New Journal of Chemistry

### Electronic Supplementary Information

# Insights into the structure and thermal stability of uranyl aluminate nanoparticles

Tony Chave, Xavier Le Goff, Andreas C. Scheinost, and Sergey I. Nikitenko

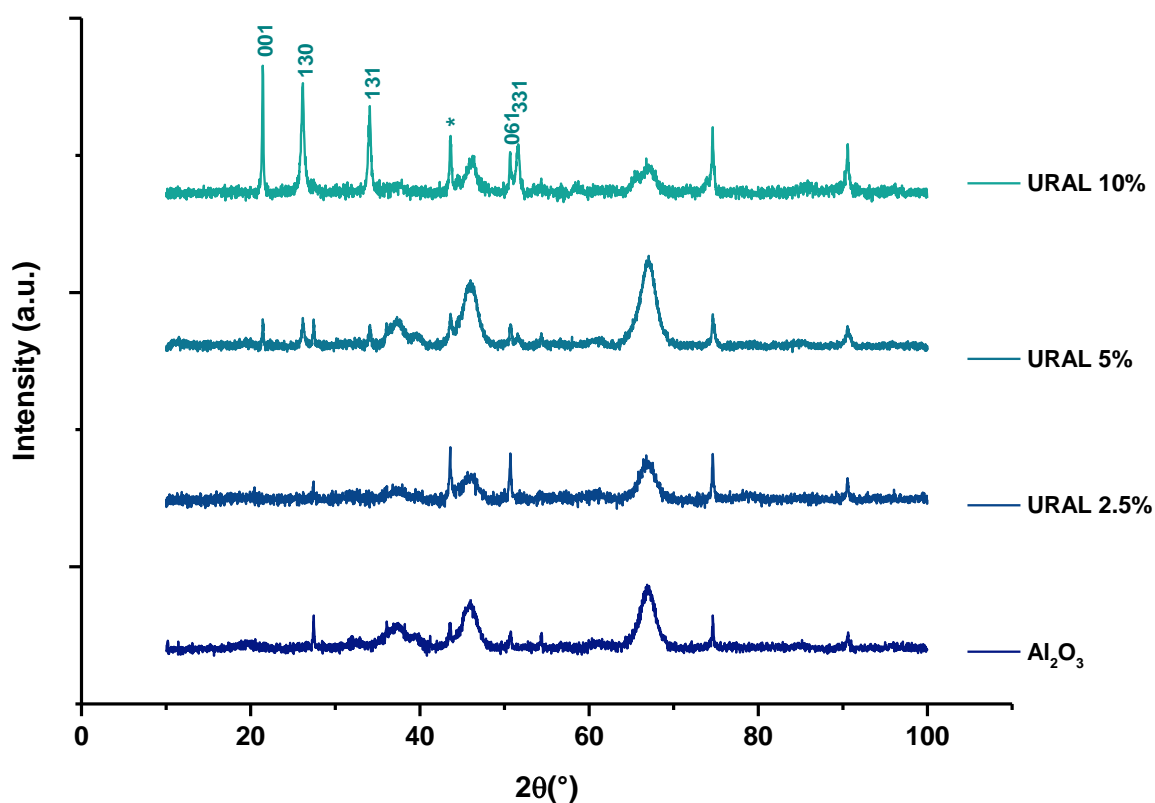


Fig. S1 XRD patterns obtained for URAL samples from 0 to 10% of added uranium after ultrasonically assisted uranium precipitation and calcination at  $800^{\circ}\text{C}$ .  $\alpha$ - $\text{U}_3\text{O}_8$  main indices of lattice planes (PDF 00-047-1493) are given as labels on URAL 10% diffraction diagram.

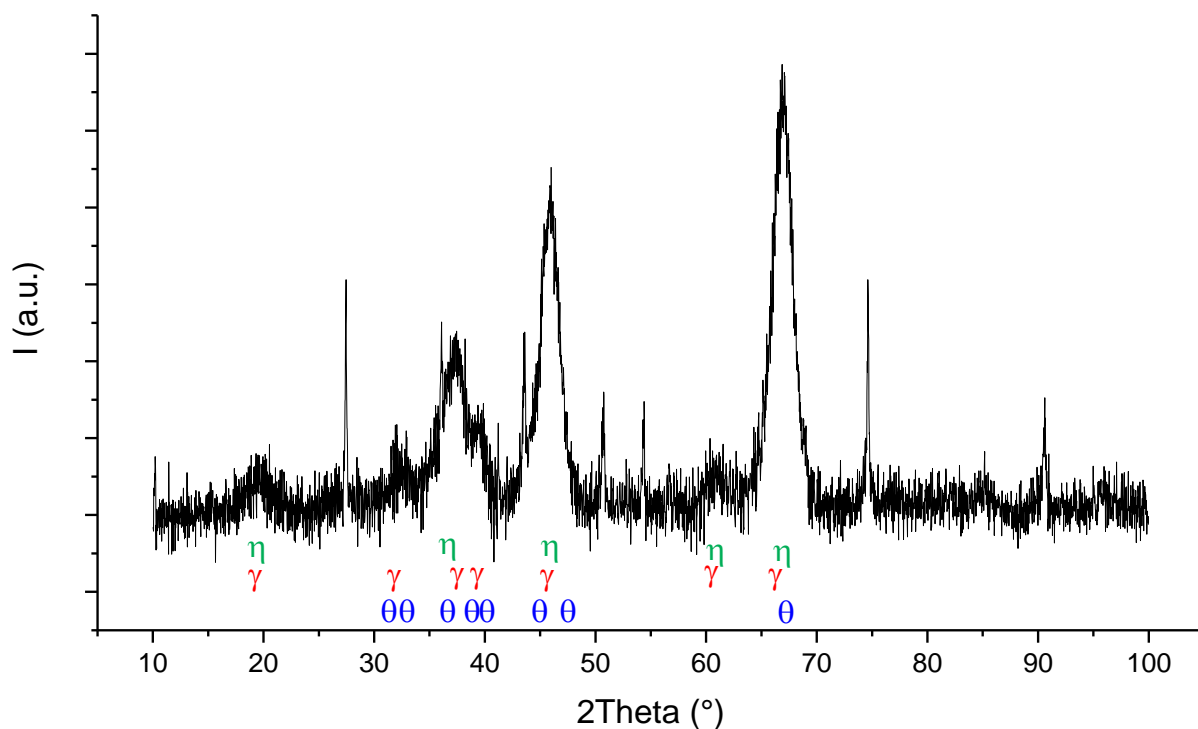


Fig. S2 XRD patterns of  $\text{Al}_2\text{O}_3$  calcined at  $800^\circ\text{C}$  after synthesis procedure. Main diffraction peaks of  $\theta\text{-Al}_2\text{O}_3$  (PDF 00-023-1009),  $\gamma\text{-Al}_2\text{O}_3$  (PDF 00-050-0741) and  $\eta\text{-Al}_2\text{O}_3$  (PDF 01-079-1557) are given for comparison.

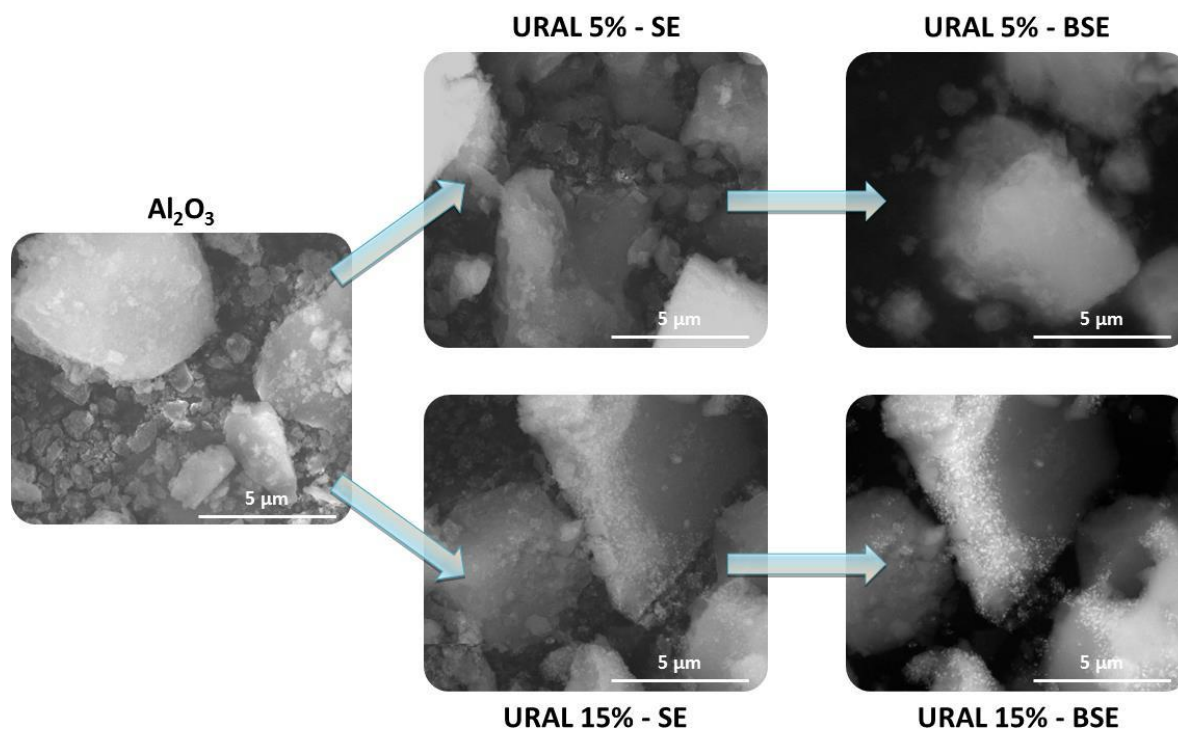


Fig. S3 Secondary electron (SE) and back-scattered electron (BSE) SEM images of URAL samples obtained without addition of uranium ( $\text{Al}_2\text{O}_3$ ) and in presence of respectively 5% and 15% of uranium.

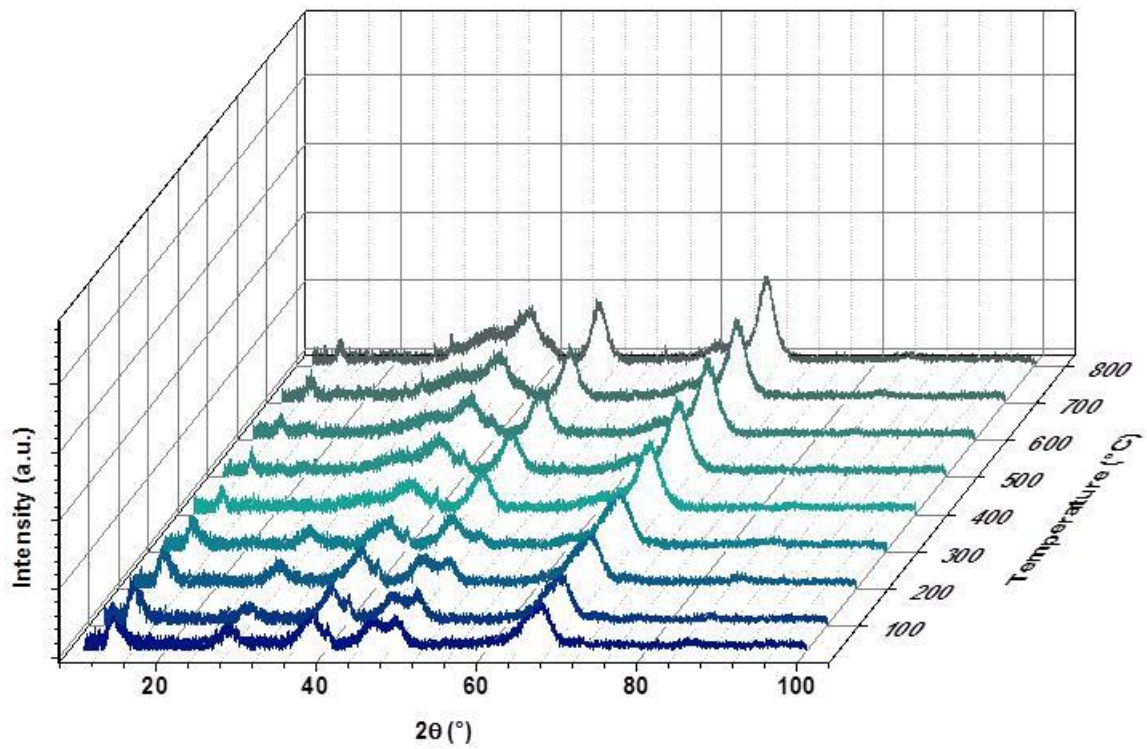


Fig. S4 XRD pattern evolution in temperature of URAL 5% sample prior calcination starting from 30°C to 800°C (from bottom to top)

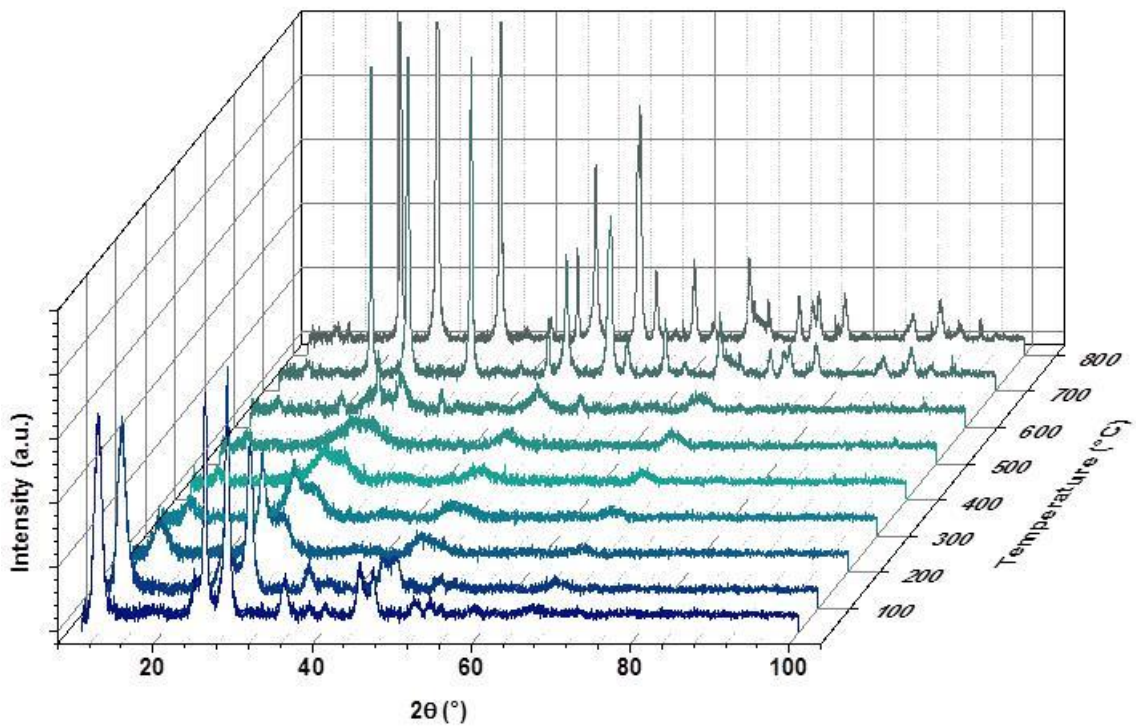


Fig. S5 XRD pattern evolution in temperature of URAL 25% sample prior calcination starting from 30°C to 800°C (from bottom to top).

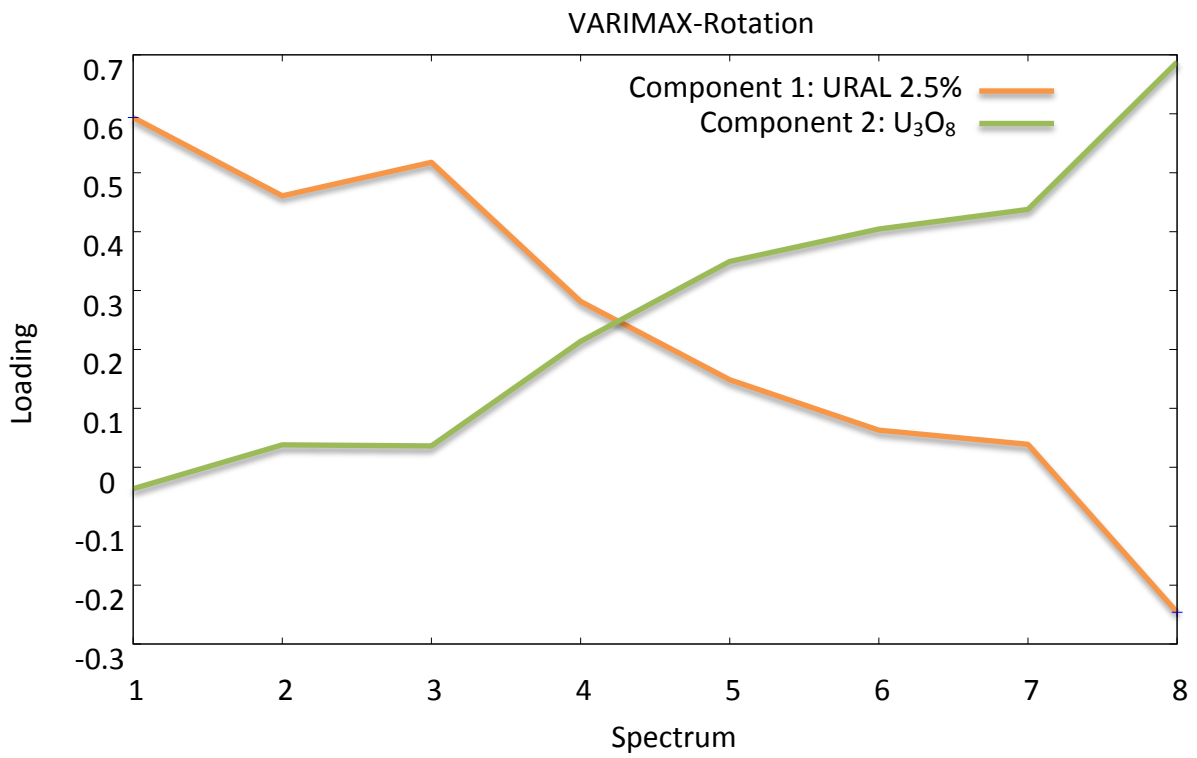


Fig. S6 Loadings of component 1 and 2 for the EXAFS spectra of samples URAL 2.5 to 25% (Spectra 1 to 7) with URAL 25% 500°C (Spectrum 3) and of U<sub>3</sub>O<sub>8</sub> (spectrum 8).

**Appendix: Diffraction data of observed phases**

**Table S1 XRD PDF data sheets with d-spacings, relative intensities and  $2\theta$  (Cu K $\alpha$ ) as well as indices of lattice planes are given for  $\alpha$ -U<sub>3</sub>O<sub>8</sub>, ammonium diuranate and  $\beta$ -UO<sub>3</sub>**

<b><math>\alpha</math>-U<sub>3</sub>O<sub>8</sub> (PDF 00-047-1493)</b>					
<b>d(Å)</b>	<b><math>2\theta</math></b>	<b>Intensity</b>	<b>h</b>	<b>k</b>	<b>l</b>
4.146	21.42	94	0	0	1
3.428	25.97	100	1	3	0
3.358	26.52	56	2	0	0
2.642	33.90	74	1	3	1
2.609	34.35	37	2	0	1
2.073	43.63	19	0	0	2
1.993	45.47	14	0	6	0
1.952	46.49	22	3	3	0
1.796	50.80	16	0	6	1
1.774	51.47	27	1	3	2
1.766	51.72	50	3	3	1
1.714	53.41	14	2	6	0
1.584	58.20	17	2	6	1
1.421	65.65	17	3	3	2

<b>Ammonium Diuranate (PDF 00-044-0069)</b>					
<b>d(Å)</b>	<b><math>2\theta</math></b>	<b>Intensity</b>	<b>h</b>	<b>k</b>	<b>l</b>
7.252	12.20	100	0	0	2
3.630	24.51	22	0	0	4
3.522	25.27	60	2	2	0
3.169	28.14	70	2	2	2
2.527	35.50	18	2	2	4
2.033	45.51	10	2	2	6
1.991	44.53	10	6	0	0
1.957	46.36	11	6	0	2

<b><math>\beta</math>-UO<sub>3</sub> (PDF 01-071-2124)</b>					
<b>d(Å)</b>	<b><math>2\theta</math></b>	<b>Intensity</b>	<b>h</b>	<b>k</b>	<b>l</b>
4.810	18.43	51	2	1	0
3.583	24.83	85	0	4	0
3.438	25.90	29	1	0	1
3.404	26.16	34	0	2	1
3.100	28.78	40	1	2	1
3.075	29.02	43	3	2	0
3.029	29.46	100	-2	2	1
1.943	46.71	23	-2	6	1
1.918	47.36	27	4	2	1

Table S2 XRD PDF data sheets with d-spacings, relative intensities and  $2\theta$  (Cu K $\alpha$ ) as well as indices of lattice planes are given for  $\gamma$ -Al<sub>2</sub>O<sub>3</sub>,  $\theta$ -Al<sub>2</sub>O<sub>3</sub> and  $\eta$ -Al<sub>2</sub>O<sub>3</sub>

<b><math>\gamma</math>-Al<sub>2</sub>O<sub>3</sub>(PDF 00-050-0741)</b>					
<b>d(Å)</b>	<b>2<math>\theta</math></b>	<b>Intensity</b>	<b>h</b>	<b>k</b>	<b>l</b>
4.584	19.35	50	1	1	1
2.807	31.86	75	2	2	0
2.394	37.54	90	3	1	1
2.292	39.28	30	2	2	2
1.985	45.67	100	4	0	0
1.621	56.75	50	4	2	2
1.528	60.55	75	5	1	1
1.403	66.60	100	4	4	0
1.146	84.47	30	4	4	4

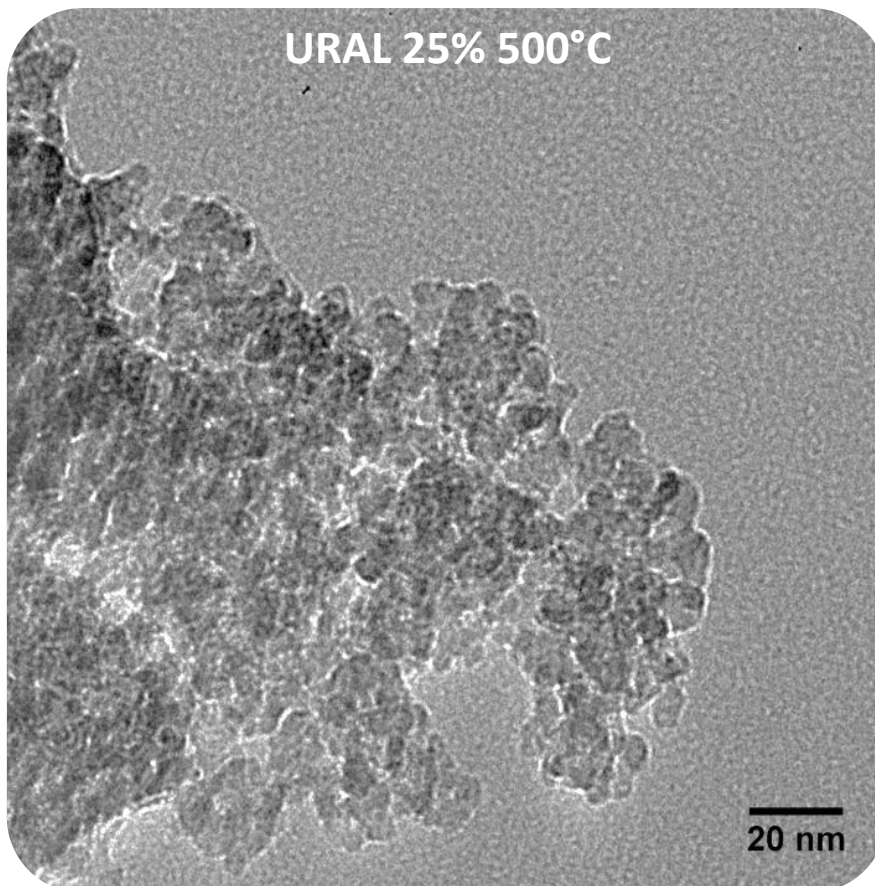
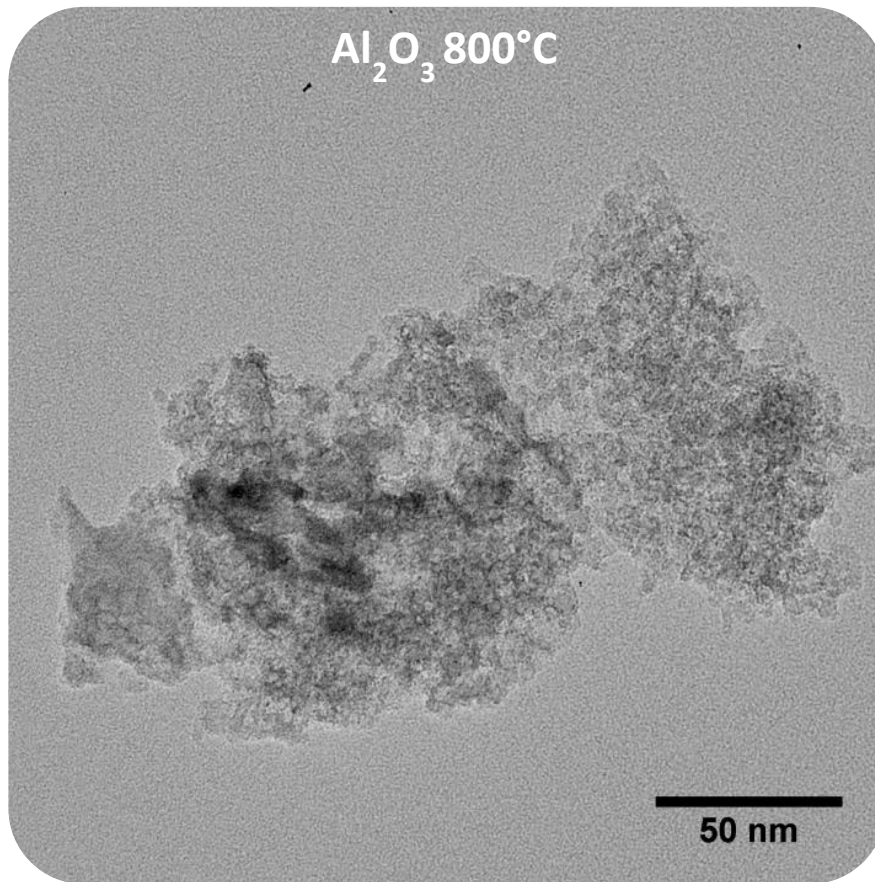
  

<b><math>\theta</math>-Al<sub>2</sub>O<sub>3</sub>(PDF 00-023-1009)</b>					
<b>d(Å)</b>	<b>2<math>\theta</math></b>	<b>Intensity</b>	<b>h</b>	<b>k</b>	<b>l</b>
2.837	31.51	80	-4	0	1
2.730	32.78	65	0	0	2
2.444	36.74	60	1	1	1
2.315	38.87	45	4	0	1
2.257	39.91	35	2	0	2
2.019	44.86	45	-1	1	2
1.909	47.59	30	6	0	0
1.543	59.91	25	-3	1	3
1.488	62.34	25	1	1	3
1.453	64.05	25	0	2	0
1.388	67.40	100	4	0	3

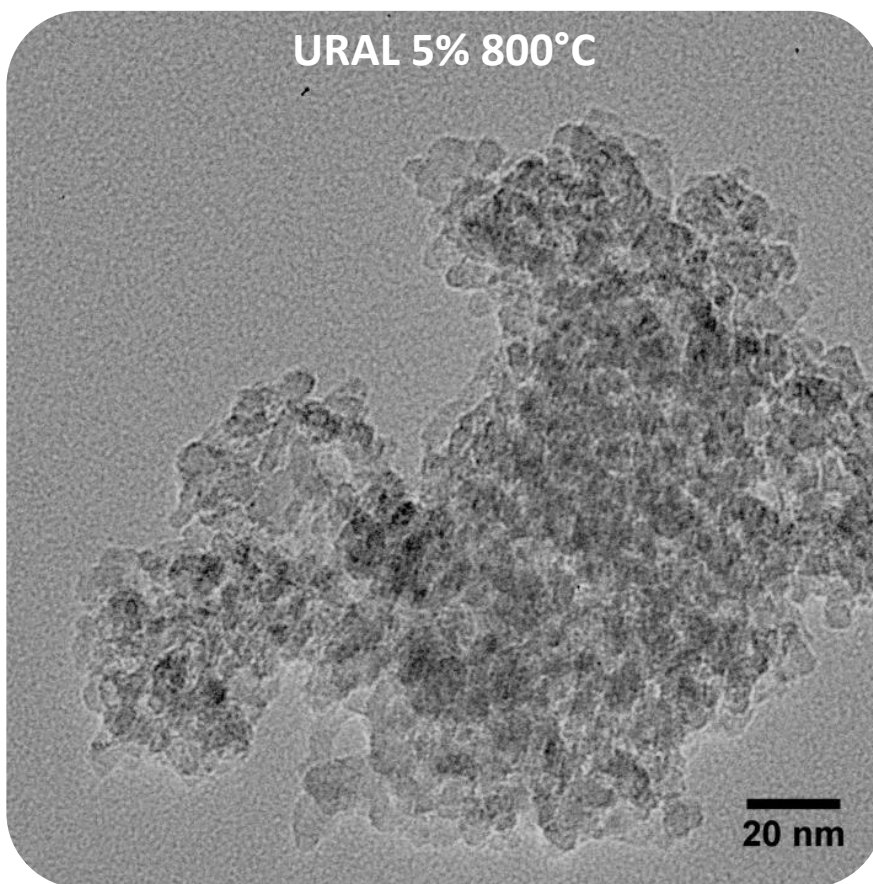
  

<b><math>\eta</math>-Al<sub>2</sub>O<sub>3</sub>(PDF 01-079-1557)</b>					
<b>d(Å)</b>	<b>2<math>\theta</math></b>	<b>Intensity</b>	<b>h</b>	<b>k</b>	<b>l</b>
4.569	19.41	43	1	1	1
2.386	37.67	44	3	1	1
2.285	39.41	18	2	2	2
1.979	45.83	76	4	0	0
1.816	50.21	11	3	3	1
1.523	60.76	22	5	1	1
1.399	66.82	100	4	4	0
1.338	70.32	12	5	3	1
0.885	121.05	10	8	4	0
0.808	144.98	23	8	4	4

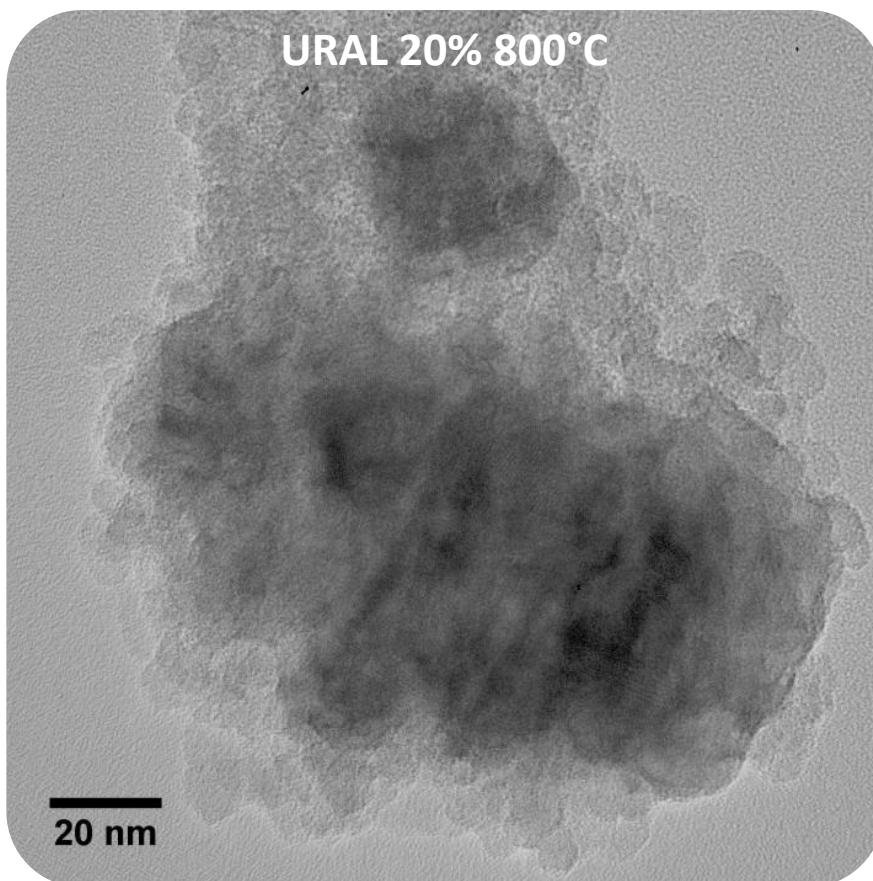
Appendix: Full size TEM images



URAL 5% 800°C

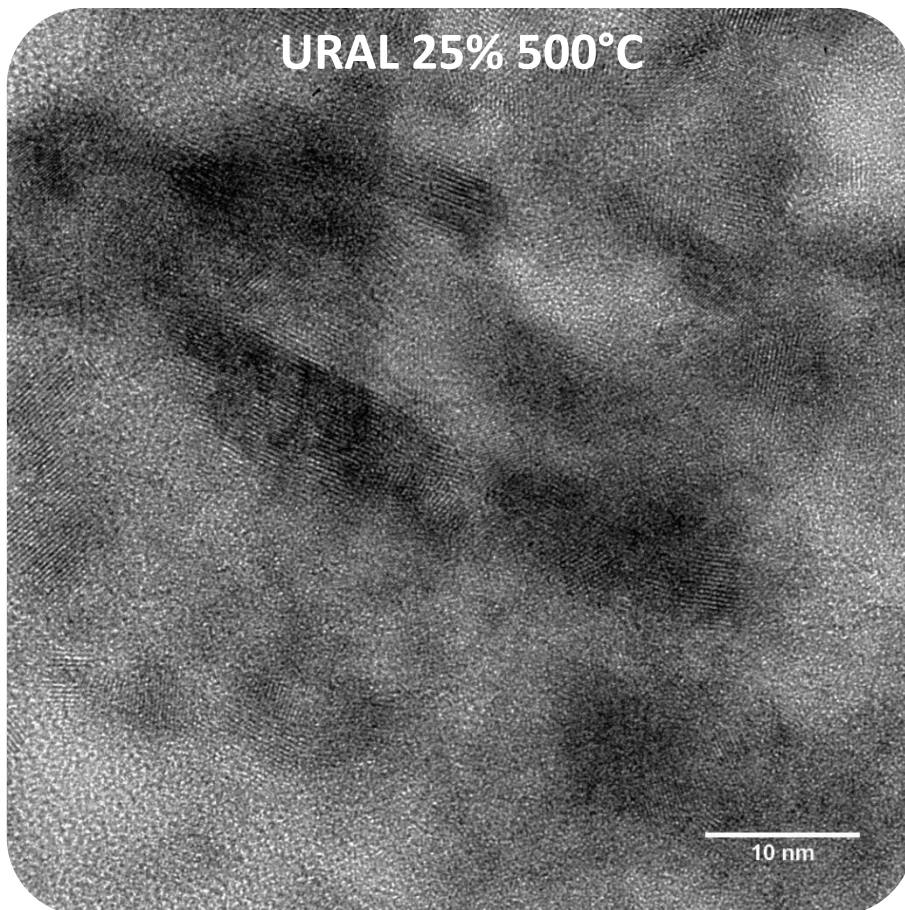


URAL 20% 800°C





URAL 25% 500°C



URAL 5% 800°C

