

Supplementary material

Recovery of niobium and tantalum by solvent extraction from Sn-Ta-Nb mining tailings

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Supplemental Table S1. Previous studies to determine the optimum leaching solution [17]

Leaching solution	[Nb] (g/L)	[Ta] (g/L)
[HF]= 4 N	1.72	2.85
[H ₂ SO ₄]= 8 N	40 ppm	80 ppm
[HF]= 4 N/[H ₂ SO ₄]= 8 N	1.62	2.72
[HF]= 6 N/[H ₂ SO ₄]= 8 N	1.90	3.27

Supplemental Table S2. Composition of the insoluble residues at different s/l ratios.

% wt.	Insoluble residue						
	Slag/leaching solution ratio (g/L)						
	20	30	40	50	60	75	100
Ta ₂ O ₅	6.40	6.43	5.49	9.75	9.56	5.49	6.54
Nb ₂ O ₅	5.63	6.55	4.50	7.82	7.79	4.46	5.23
SiO ₂	2.39	2.15	1.30	3.44	4.97	5.19	3.95
SnO ₂	6.57	6.31	8.87	9.24	8.84	7.47	7.89
CaO	30.43	31.28	35.72	31.02	31.74	35.80	38.06
MnO	0.93	1.10	0.46	0.76	1.16	0.73	0.58
Fe ₂ O ₃	0.72	0.80	0.37	0.51	0.76	0.46	0.43
ZrO ₂	16.79	13.86	11.38	8.06	6.66	4.94	2.63
Al ₂ O ₃	5.98	8.08	11.32	10.48	9.45	8.84	8.31
Na ₂ O	2.02	1.19	4.49	2.06	1.34	5.72	1.38
P ₂ O ₅	1.47	1.45	1.23	1.42	1.45	1.18	1.18
CeO ₂	4.46	3.95	2.85	2.57	2.25	2.19	3.12
TiO ₂	0.24	0.25	0.17	0.34	0.28	0.16	0.23
Y ₂ O ₃	2.17	3.98	1.61	1.42	1.27	1.24	1.53
La ₂ O ₃	2.14	1.92	1.39	1.26	1.06	1.09	1.52
Nd ₂ O ₃	3.16	2.91	2.20	1.93	1.67	1.58	2.28
K ₂ O	0.13	0.17	0.21	0.23	0.17	0.14	0.04
HfO ₂	0.64	0.64	0.60	0.42	0.34	0.28	0.11
Gd ₂ O ₃	1.10	0.94	0.79	0.69	0.61	0.57	0.79
SO ₃	4.82	5.00	4.77	5.80	7.36	13.09	12.49
Sm ₂ O ₃	0.68	0.62	0.56	0.46	0.46	0.42	0.50
BaO	0.16	0.13	0.15	0.18	0.12	0.12	0.10
Pr ₂ O ₃	0.54	0.50	0.40	0.34	0.37	0.30	0.43
ThO ₂	1.18	1.08	0.80	0.70	0.63	0.60	0.81

U	0.55	0.48	0.44	0.38	0.33	0.35	0.33
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Supplemental Table S3. Insoluble residue for each s/l ratio.

Slag/leaching solution ratio (g/L)	Insoluble residue (% wt.)
20	30.40
30	34.75
40	28.38
50	22.70
60	32.08
75	41.14
100	38.63

Supplemental Table S4. Nb Precursor precipitation conditions.

Sample	[Nb] ₀ (g/L)	Nb sol.:NH ₃	Molar ratio (n _{Nb} /n _{NH₃})	[Nb] _f (g/L)	Yield (%)
Nb_1	6.53	5:1	0.067	0.0942	97.2
Nb_2	5.41	5:1	0.056	0.0094	98.3
Nb_3	4.82	3:1	0.030	0.0017	99.7
Nb_4	6.20	20:17	0.015	0.0148	99.8

Supplemental Table S5. Ta precursor precipitation conditions.

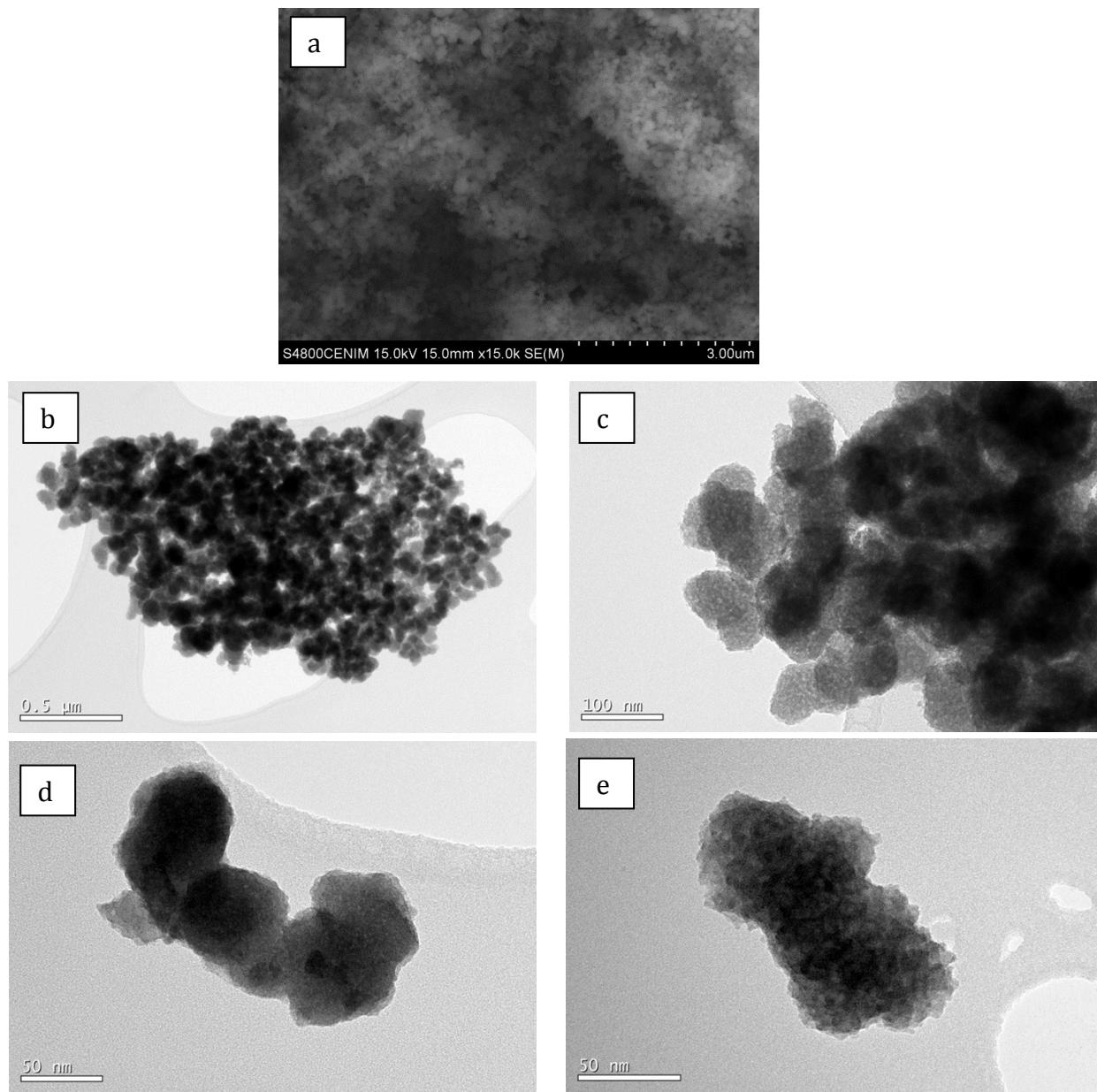
Sample	[Ta] ₀ (g/L)	[KF] (g/L)	Ta sol.:KF	Molar ratio (n _{Ta} /n _{KF})	[Ta] _f (g/L)	Yield (%)
Ta_1	9.30	30	5:3	0.152	2.17	74.5
Ta_2	9.30	60	2:1	0.008	1.16	86.4
Ta_3	7.53	60	4:3	0.054	0.28	96.3
Ta_4	8.24	90	3:1	0.088	0.65	92.1
Ta_5	7.01	90	3:2	0.038	1.36	80.6
Ta_6	7.63	90	1:1	0.027	0.29	96.3
Ta_7	9.43	90	1:2	0.017	0.23	97.6

Supplemental Table S6. Chemical composition of calcined and purified precursor determined by XRF.

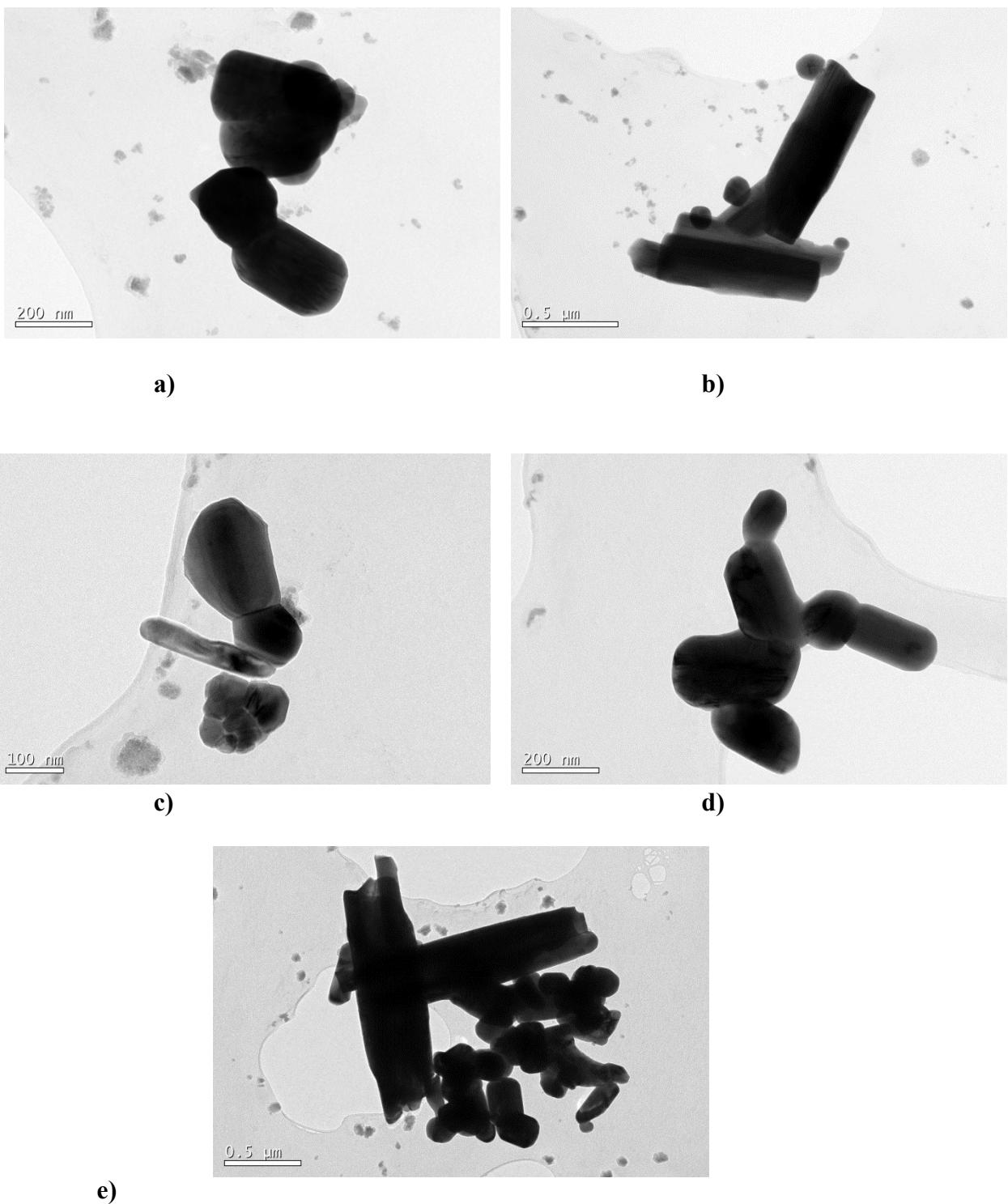
Oxide (% wt.)	Calcined precursor	Calcined precursor purified
Ta ₂ O ₅	82.01	97.31
K ₂ O	15.19	-
Nb ₂ O ₅	1.67	1.64
CaO	0.59	0.52
SnO ₂	0.45	0.19
Fe ₂ O ₃	0.05	-

NiO	0.02	-
ZnO	0.01	-

Supplemental Figure S1. $\text{Nb}_2\text{O}_5\text{.nH}_2\text{O}$ nanoparticles observed by SEM (a) and by TEM (b-e).



Supplemental Figure S2. TEM images of Nb_2O_5 morphologies.



Supplemental Figure S3. SEM images of Ta precursor.

