

Dissolution Studies on Large, high-fired BeO Ceramic Particles using Ammonium Bifluoride Aqueous Solutions

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Overview

- BeO Particle size and material used.
- Experimental details.
- Dissolution variables that affect recoveries.
- Impact of particle size on dissolution.
- Media spiked with large particles.
- Conclusions.

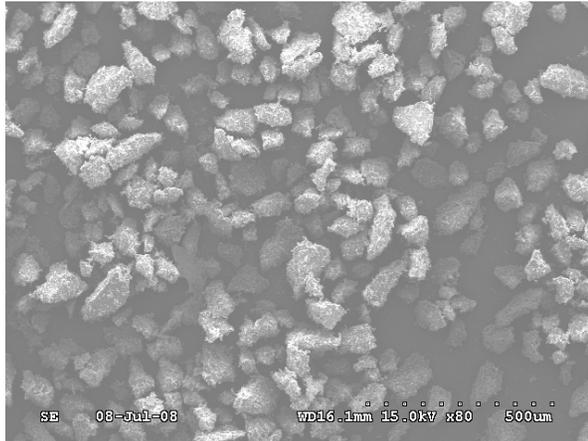
How Large should the Particles be from a Safety Perspective? (BeO Particle Size [spherical] and Amount of Beryllium)

Particle size (diameter, cm)	Particle size, (diameter, μm)	Volume, (cc)	Beryllium oxide mass, (μg)	Beryllium mass, (μg)
0.0001	1	5.24E-13	0.000002	5.65E-07
0.001	10	5.24E-10	0.002	0.00057
0.005	50	6.55E-08	0.20	0.071
0.01	100	5.24E-07	1.57	0.57
0.02	200	4.19E-06	12.57	4.52
0.1	1000	5.24E-04	1570.8	565.5

850 CFR- DOE action limits at 0.2μg/100cm² or m³

BeO Details

- Samples used:
- Thermalox® BeO. $\rho=2.88$ g/cm³(BeO theoretical 3.01).
- Ceramic prepared by sintering UOX at 1550°C for 4 hours.
- Ceramic block pulverized by impact grinding and size fractionated using sieves.

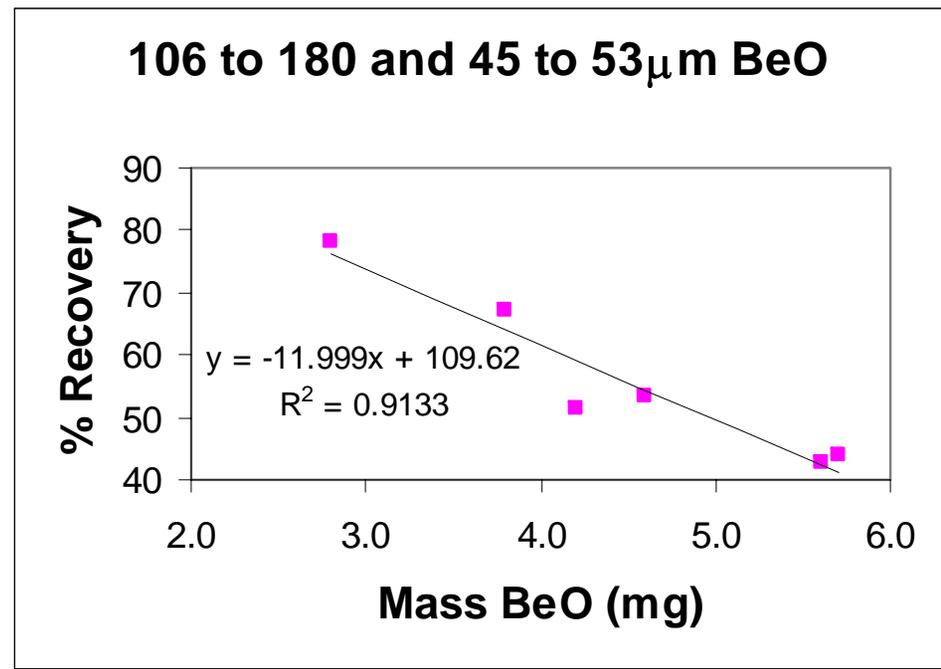
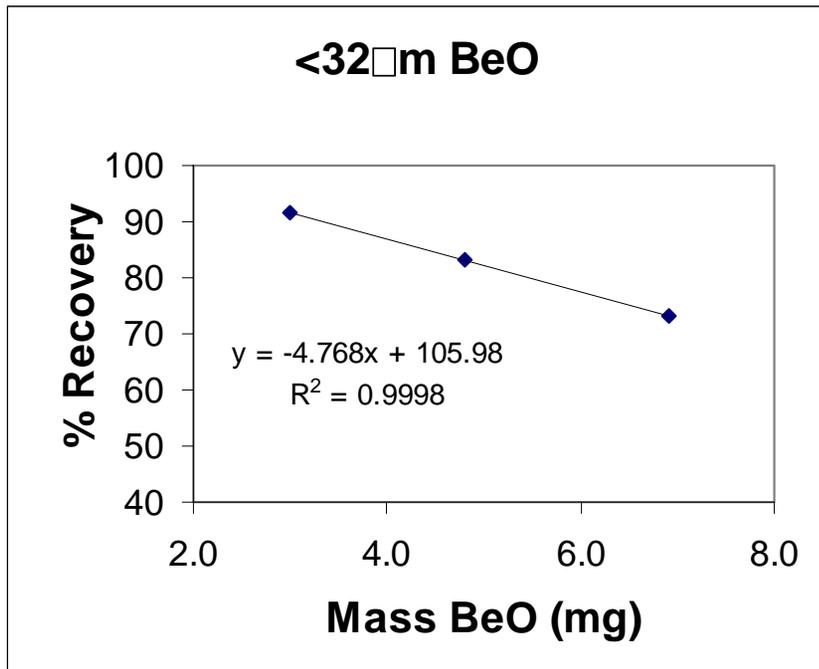


Mesh size	Particle size, μm
-500	<32
-270 +325	45 to 53
-270 +140	53 to 90
-140 +270	90 to 106
-70 +80	180 to 212

Experimental Details

- Dissolution solution 1% or 3% ABF in water (wt/vol).
- Dissolution at elevated temperatures was done by placing sealed plastic sample tubes in a gravity convection oven.
- All samples analyzed by fluorescence following 20x dilution (1 part dissolution solution and 19 parts HBQS dye solution as in NIOSH 7704 or ASTM 7202).
- Excitation wavelength 365 and emission at 475nm.

Effect of Amount of Sample on Recovery



1%ABF-5ml, 24 hours at 90°C, 15ml falcon tubes

Effect of Tube Shape/Size on Dissolution

Dissolution conditions: 3% ABF 5ml, 90°C

Sample type: 180-212 μ m fraction

Sample amount: 1.1 to 1.6 mg

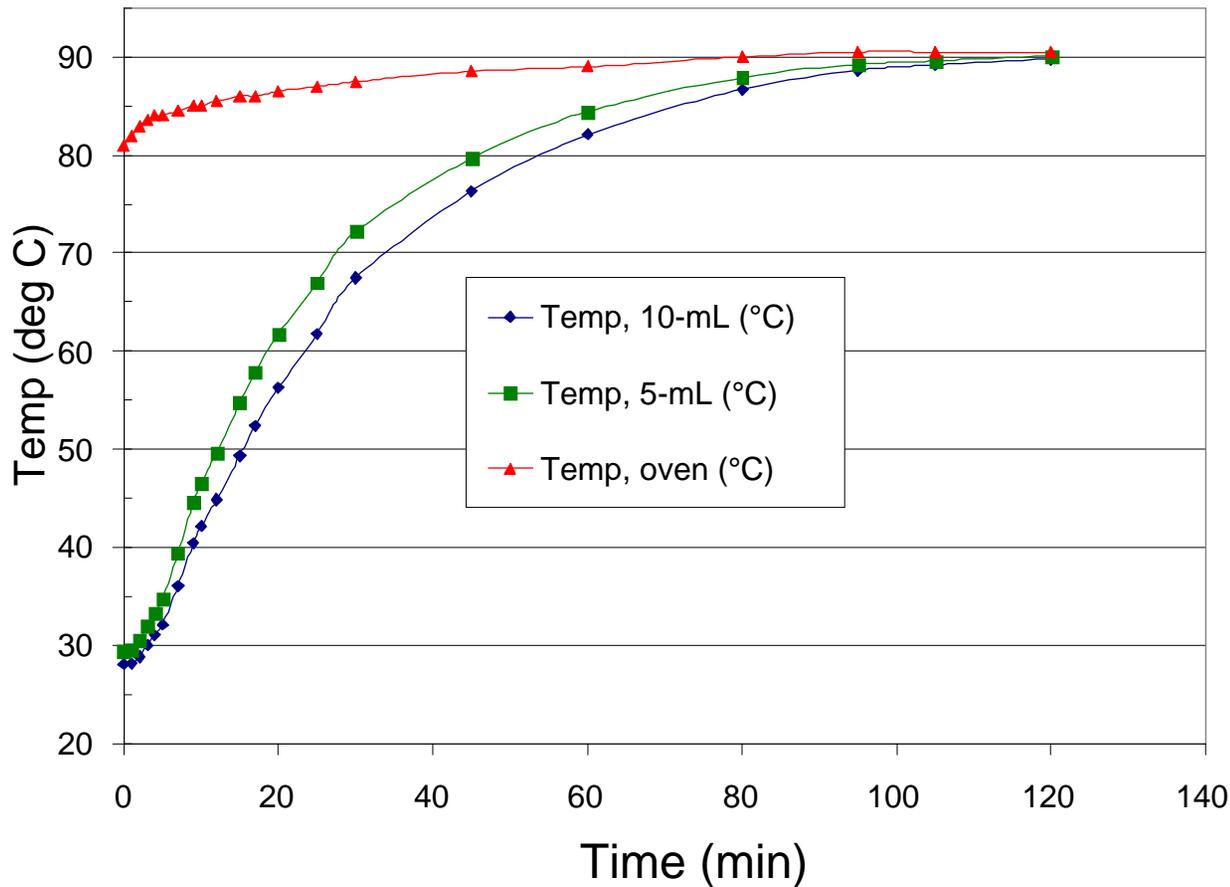
	Recovery %		
Dissolution time, hrs	15ml Falcon tube	50ml falcon tube	10ml round bottom tube
2hrs	36 \pm 4	48 \pm 3	50 \pm 4
4hrs	67 \pm 5	94 \pm 9	100 \pm 5

All uncertainties based on standard deviations for n=3

Temperature-Time Profile of Samples in the Oven used for Dissolution

Data for

- 50ml Falcon Tubes with water, sealed
- Gravity convection oven pre-heated to 90°C



Dissolution Conditions

- Vessel used: 50ml falcon tube or 120ml cup
- Sample size: 1-2mg of BeO
- ABF concentration in water: 1 or 3%
- ABF volume: 5, 10, 15 or 50ml
- Oven: Preheated, gravity convection
- Dissolution time >2 hours or more
- Dissolution Temperature: 60-90C

Dissolution in 1%ABF at 90°C

	Percent Recovery in 1% ABF at 90°C (Std Deviation on three samples)			
Time, (hr)	BeO Size			
	<32μm	45 to 53μm	90 to 106μm	180-212μm
2	63 (±2)	35 (±3)	26 (±1)	19 (±1)
4	90 (±6)	66 (±8)	53 (±1)	48 (±1)
6		85 (±1)	79 (±8)	74 (±5)
8		90 (±5)	98 (±2)	96 (±3)

5ml ABF in 50ml falcon tube

Dissolution in 3%ABF at 90°C

	Percent Recovery in 3% ABF at 90°C			
Time (hr)	BeO Size			
	<32μm	45 to 53μm	90 to 106μm	180-212μm
2	89 (2)	64 (5)	55 (2)	41 (4)
4	95 (5)	106 (1)	102 (12)	97 (5)

5ml ABF in 50ml falcon tube

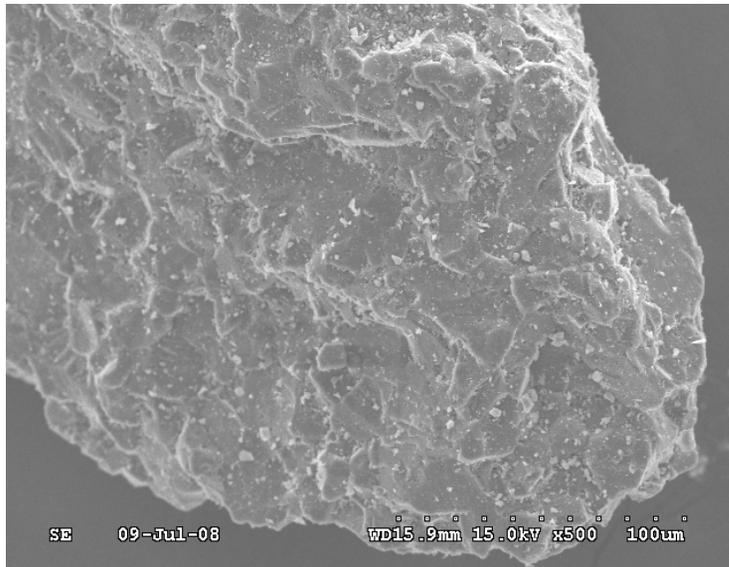
Dependence of Time and Temperature on dissolution in 3%ABF (5ml ABF in 50ml Falcon tubes)

	Percent Recovery											
Time	BeO Size											
(hr)	<32 μ m				90 to 106 μ m				180-212 μ m			
	25 °C	60 °C	80 °C	90 °C	25 °C	60 °C	80 °C	90 °C	25 °C	60 °C	80 °C	90 °C
2	19	52	81	89	3.9	23	55	55	2.6	16	28	41
4	26.5	75	93	95	6.8	47	91	102	5.8	31	61.3	97
6	34	83			9.8	65			7.9	46	91	
8	45	90			13.4	84			9.6	67		
10						95				84		
12	57				19.3				13	91		
24	72				29				22			

Cubic dependence of time on particle size is not seen

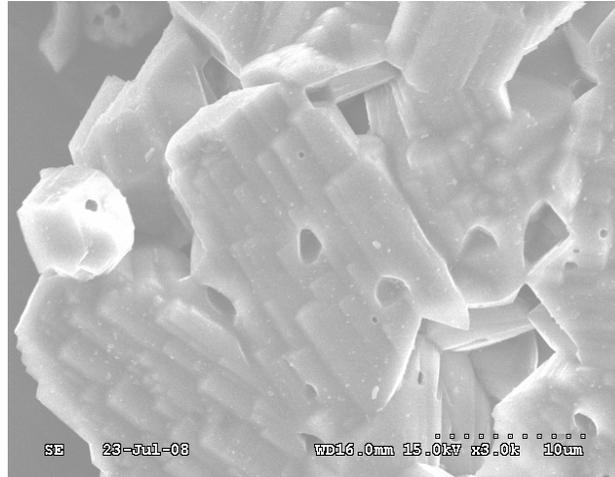
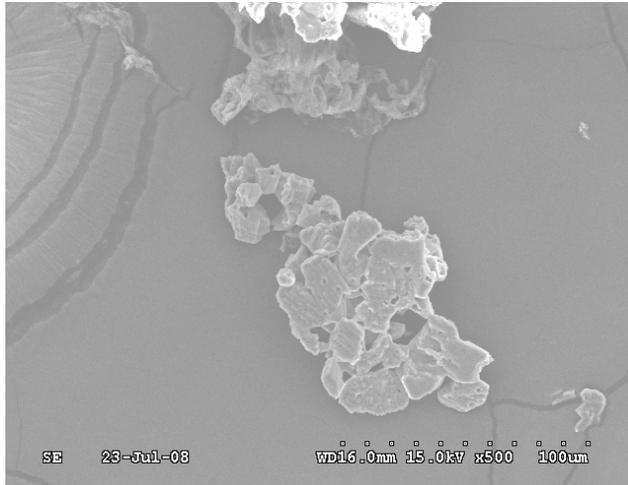
Particles as Received

(180-212 μ m)

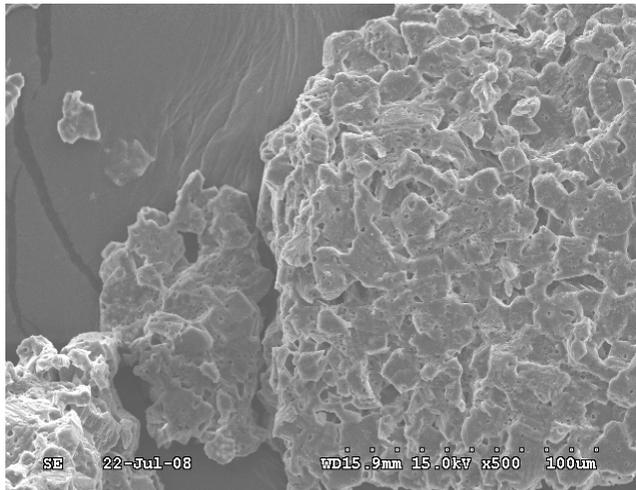


Partially Dissolved Particles

(After 1 hour in 1%ABF at 90°C)



90 to 106 μm



180 to 212 μm

Dissolution of BeO on Media

	Percent Recovery on Media in 3%ABF at 90C for 5 hours			
	Filter / Wipe Medium			
BeO Size	MCE*	Whatman 541*	Palintest**	Cotton Glove#
<32 μ m	92 (6)	92 (5)	89 (6)	111 (16)
90 to 106 μ m	100 (7)	99 (2)	94 (5)	98 (12)
180 to 212 μ m	92 (1)	96 (3)	98 (5)	90 (6)
* 10ml ABF in 50ml falcon tube, **15ml ABF in 50 ml falcon tube, #50ml ABF in 120ml polypropylene cup with lid				

Effect of Dissolution in Presence of Metal Working Fluid (MWF)

Percent Recovery on Cotton Gloves		
3%ABF at 90°C for 5hrs		
No MWF	Virgin MWF	Used MWF
98(12)	92 (3)	91(4)

BeO size 90 to 106 μ m,

1ml in MWF with 50ml ABF solution in 120ml PP cup with lid

Conclusions

- The choice of sample tube and the amount of sample (BeO) used impact dissolution.
- Temperature effects are very important.
- Dissolution is speeded up by using more concentrated ABF solution.
- The influence of particle size on dissolution time is not strong, due to the porosity in ceramic BeO .
- Dissolution procedures are effective in presence of a variety of media and also when metal working fluids are present.