

Supporting Information

Temperature and Atmosphere-Driven Phase Evolution in Sol-Gel Synthesized Barium Nickelate Powders

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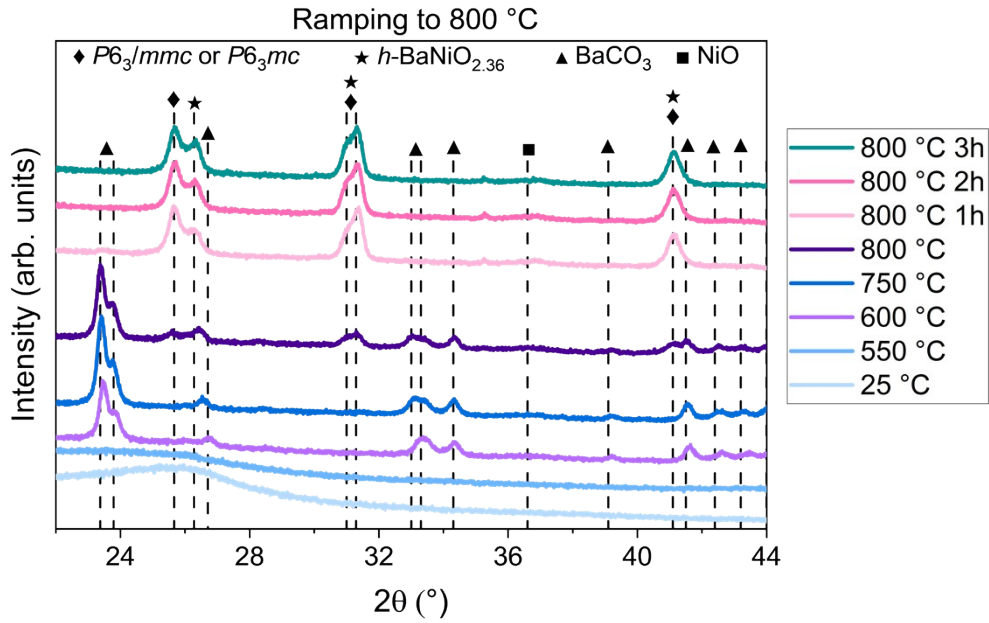


Figure S1. In-situ XRD of a BNO dried sol-gel. Gel was ramped to 800 °C at 7 °C/min under 80 mL/min of flowing air with a dwell time of 3h at 800 °C. BNO $P6_3/mmc$ and $P6_3mc$ diffraction peaks are labeled with ◆, BNO $h\text{-BaNiO}_{2.36}$ diffraction peaks labeled with ★, BaCO_3 diffraction peaks are labeled with ▲, and NiO diffraction peaks are labeled with ■.

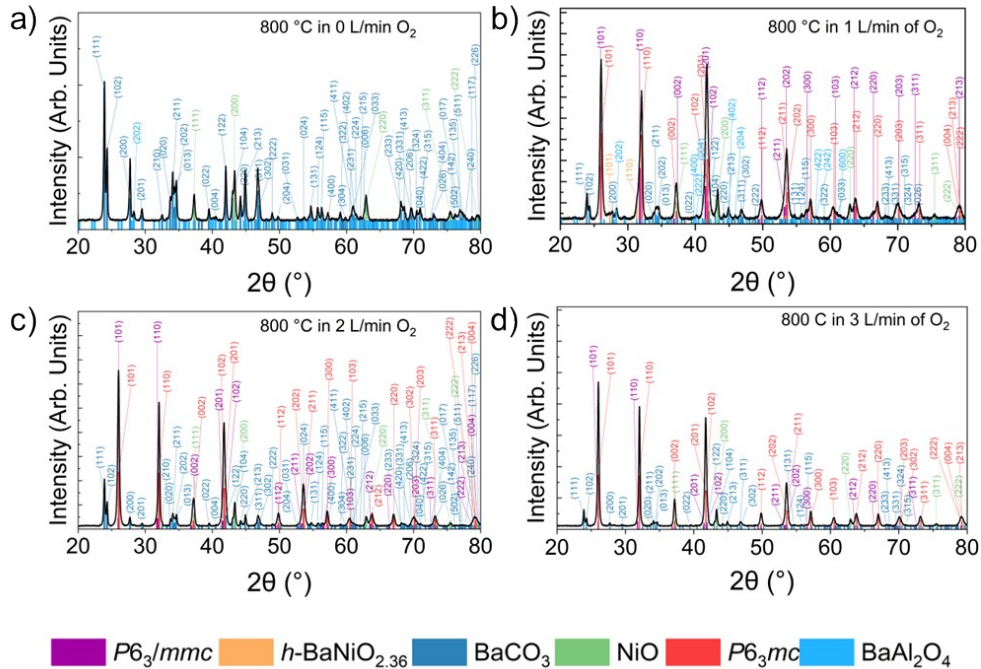


Figure S2. XRD patterns of BNO sol-gels that were calcined at temperatures ranging from 800-1000 °C and oxygen flow rates of 0-3 L/min. a) Calcined at 800 °C and 0 L/min of flowing

oxygen. b) Calcined at 800 °C and 1 L/min of flowing oxygen. c) Calcined at 800 °C and 2 L/min of flowing oxygen. d) Calcined at 800 °C and 3 L/min of flowing oxygen.

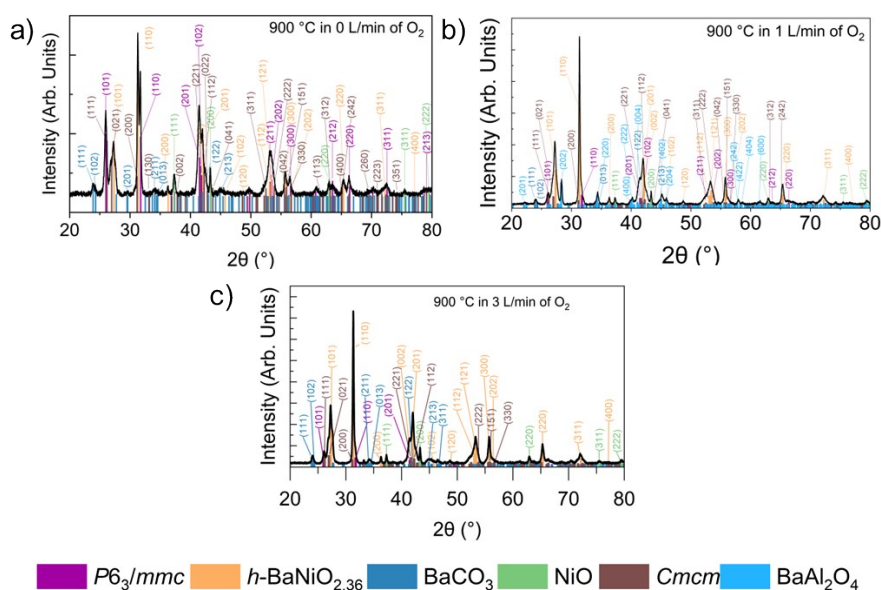


Figure S3. XRD patterns of BNO sol-gels that were calcined at 900 °C under oxygen flow rates of 0, 1, and 3 L/min. a) Calcined at 900 °C under 0 L/min of flowing oxygen. b) Calcined at 900 °C under 1 L/min of flowing oxygen. c) Calcined at 900 °C under 3 L/min of flowing oxygen.

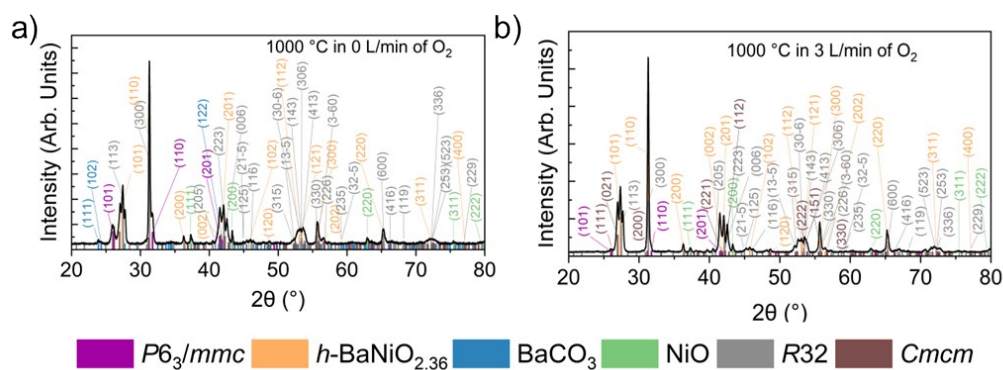


Figure S4. XRD patterns of BNO sol-gels that were calcined at 1000 °C under 0 and 3 L/min of flowing oxygen. a) Calcined at 1000 °C under 0 L/min of oxygen. b) Calcined at 1000 °C under 3 L/min of flowing oxygen.

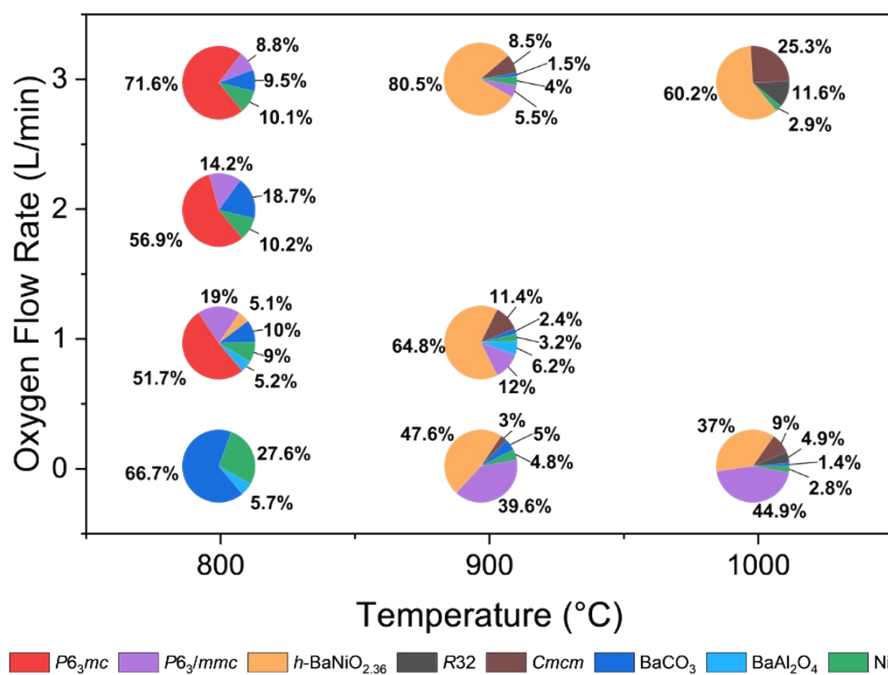


Figure S5. Rietveld refinement derived phase percentages of BNO phases and secondary phases present in the diffraction data as a function of temperature and oxygen flow rate. Each pie chart is labeled with the relative phase percentages of the BNO phases and secondary phases calculated from the diffraction data in **Figure S2**, **S3**, and **S4**.

Table S1. Rietveld refinement quantitative analysis results of BNO phases and secondary phases present in the diffraction data as a function of temperature and oxygen flow rate.

Sample	Phase percentage								Refinement factor		
	6mm	6/mmm	BNO _{2.36}	BNO ₂	R32	BaCO ₃	NiO	BaAl ₂ O ₄	R _{wp}	R _{exp}	χ^2
800 °C											
0 L/min O ₂	-	-	-	-	-	66.66%	27.58%	5.76%	3.66	2.58	2.01
800 °C											
1 L/min O ₂	51.63%	19.00%	5.14%	-	-	10.04%	9.04%	5.15%	2.74	2.5	1.2
800 °C											
2 L/min O ₂	56.90%	14.20%	-	-	-	18.68%	10.22%	-	2.01	1.44	1.95
800 °C											
3 L/min O ₂	71.57%	8.80%	-	-	-	9.52%	10.11%	-	1.76	1.89	0.87
900 °C											
0 L/min O ₂	-	39.58%	47.58%	2.99%	-	5.02%	4.83%	-	4.28	3.93	1.19
900 °C											
1 L/min O ₂	-	11.89%	64.84%	11.39%	-	2.43%	3.23%	6.22%	3.75	2.45	2.34
900 °C											
3 L/min O ₂	-	5.54%	80.53%	8.50%	-	1.45%	3.98%	-	3.33	2.25	2.19
1000 °C											
0 L/min O ₂	-	44.90%	37%	8.97%	4.88%	1.44%	2.81%	-	4.59	3.27	1.97
1000 °C											
3 L/min O ₂	-	-	60.26%	25.30%	11.58%	-	2.86%	-	5.94	2.21	7.22

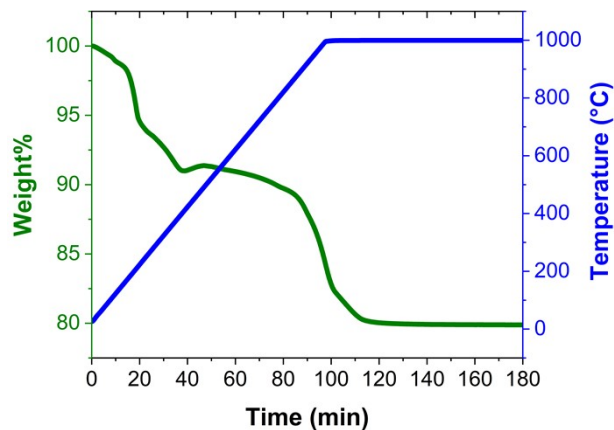


Figure S6. Thermogravimetric analysis (TGA) results collected on BNO powders that were calcined at 800 °C at 3 L/min of flowing oxygen.

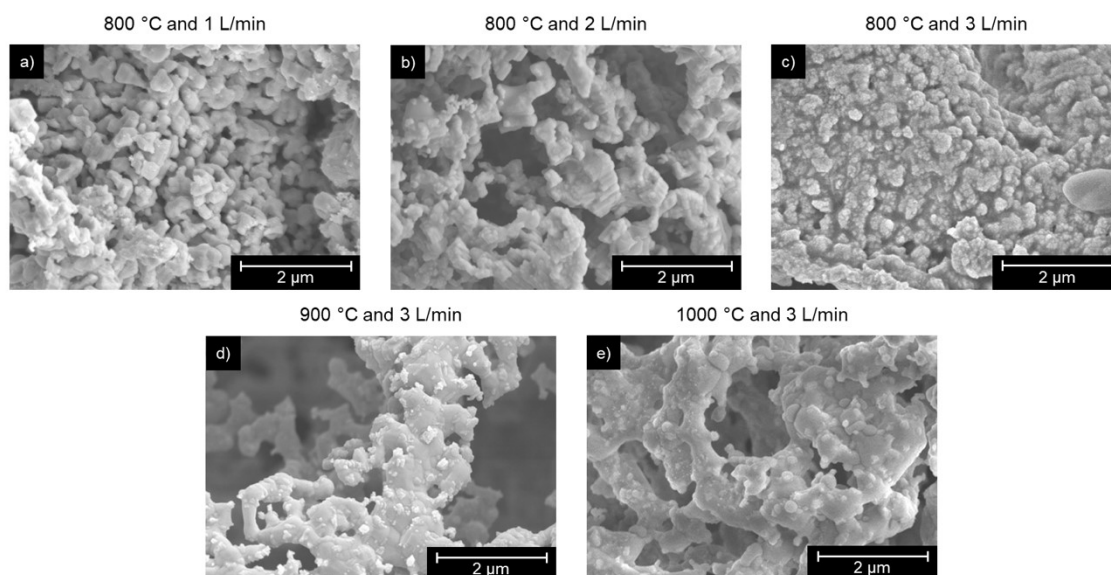


Figure S7. SEM micrographs of BNO powders illustrating the change in morphology with increasing temperature and oxygen flow rate. (a) Powder calcined at 800 °C under 1 L/min of oxygen. (b) Powder calcined at 800 °C under 2 L/min of oxygen. (c) Powder calcined at 800 °C under 3 L/min of oxygen. (d) Powder calcined at 900 °C under 3 L/min of oxygen. (e) Powder calcined at 1000 °C under 3 L/min of oxygen.

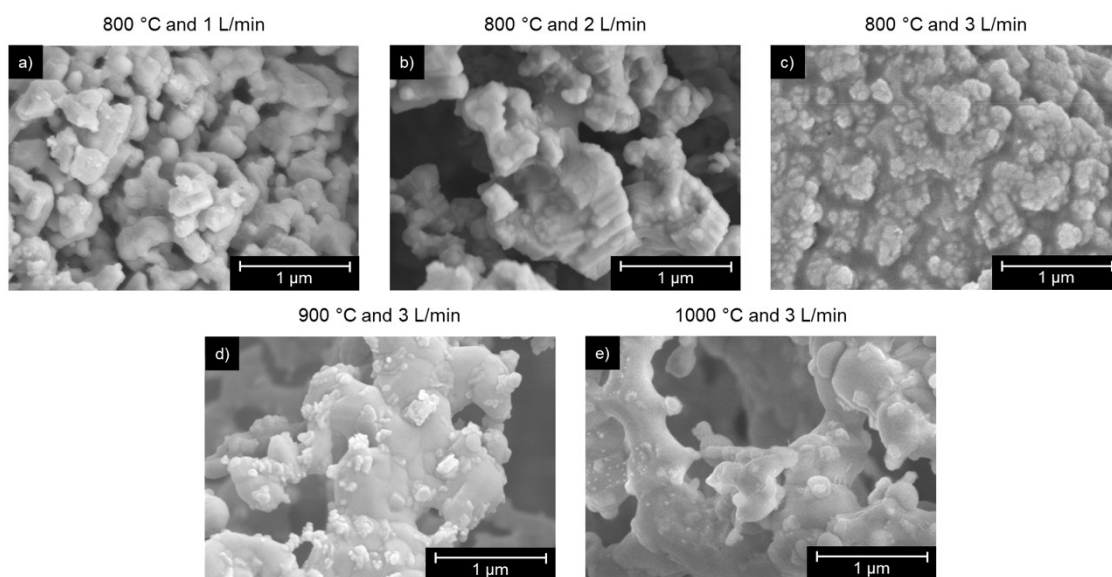


Figure S8. SEM micrographs of BNO powders illustrating the change in morphology with increasing temperature and oxygen flow rate. (a) Powder calcined at 800 °C under 1 L/min of oxygen. (b) Powder calcined at 800 °C under 2 L/min of oxygen. (c) Powder calcined at 800 °C under 3 L/min of oxygen. (d) Powder calcined at 900 °C under 3 L/min of oxygen. (e) Powder calcined at 1000 °C under 3 L/min of oxygen.

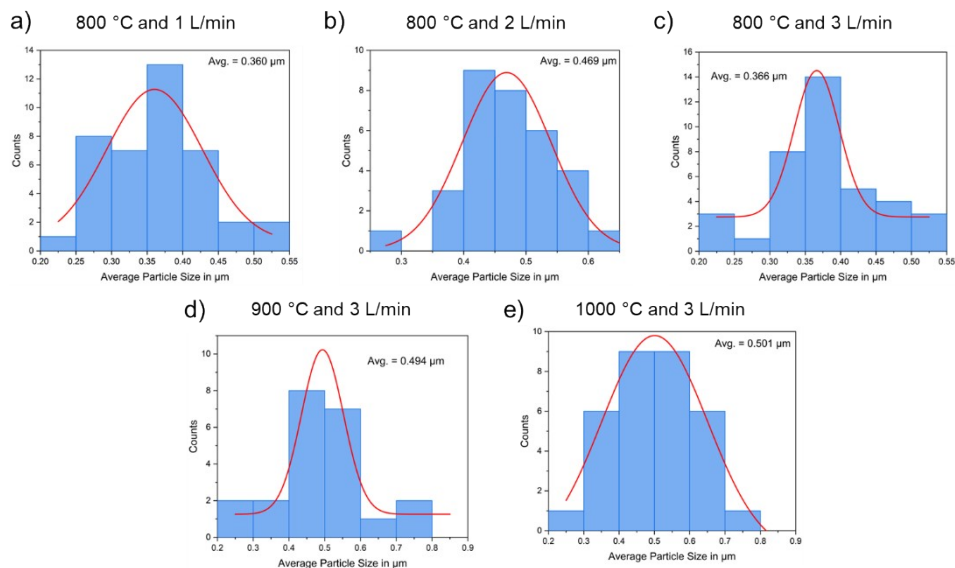


Figure S9. Average particle size of the BNO powders determined from the microscale FESEM images. (a) BNO powders calcined at 800 °C in 1 L/min of oxygen. (b) BNO powders calcined at 800 °C in 2 L/min of oxygen. (c) BNO powders calcined at 800 °C in 3 L/min of oxygen. (d) BNO powders calcined at 900 °C in 3 L/min of oxygen. (e) BNO powders calcined at 1000 °C in 3 L/min of oxygen.

Table S2. Crystallite sizes of the BNO phases as a function of temperature and oxygen flow rate. The crystallite sizes here were calculated using the Scherrer equation.

Predominant Phase	Temperature (°C)	Oxygen Flow Rate (L/min)	Crystallite Size (nm)
<i>P6₃mc</i>	800	1	23
<i>P6₃mc</i>	800	2	28
<i>P6₃mc</i>	800	3	34
<i>P6₃/mmc</i>	900	0	26
<i>h</i> -BaNiO _{2.36}	900	1	39
<i>h</i> -BaNiO _{2.36}	900	3	35
<i>P6₃/mmc</i>	1000	0	27
<i>h</i> -BaNiO _{2.36}	1000	3	39