Supporting Information

N-doped NbO_x Nanoparticle Electrocatalyst Deposited on Carbon Black for Oxygen Reduction and Evolution Reactions in Alkaline Media

Jeongsuk Seo[1,*], Won-Jin Moon[2], Wan-Gil Jung[2], and Jun-Woo Park[3]

[1] Department of Chemistry, College of Natural Sciences, Chonnam National University, 77 Yongbong-ro, Buk-gu, Gwangju 61186, Republic of Korea;

[2] Korea Basic Science Institute, Gwangju Center, 77 Yongbong-ro, Buk-gu, Gwangju 61186, Republic of Korea;

[3] Next-Generation Battery Research Center, Korea Electrotechnology Research Institute, 12 Bulmosan-ro 10beon-gil, Seongsan-gu, Changwon-si, Gyeongsangnam-do 51543, Republic of Korea

** Corresponding author: j_seo@chonnam.ac.kr*

Keywords: Bifunctional oxygen electrocatalyst, Water splitting, Nonplatinum catalyst, Niobium oxide, TaO_x nanoparticles.

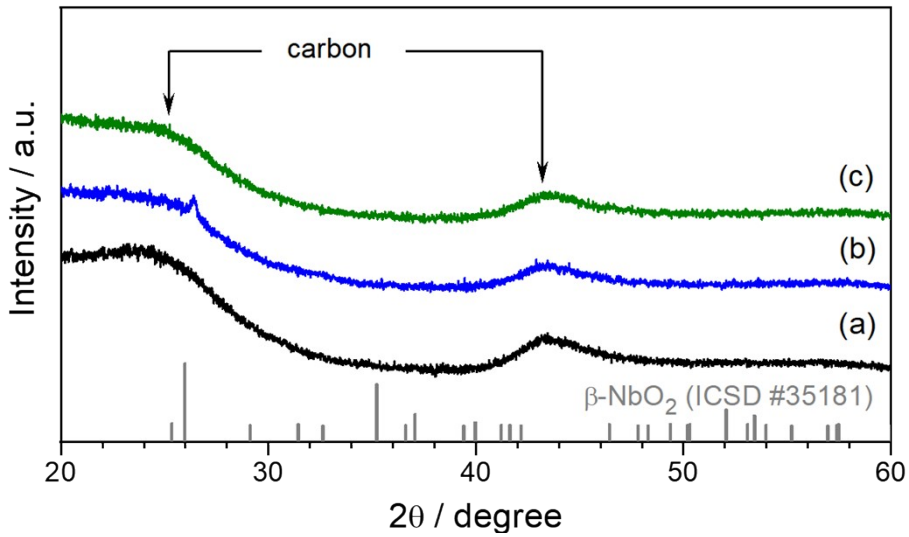

Figure S1. XRD patterns for NbO_x/CB nanoparticles (a) as-deposited prepared by electrodeposition in a non-aqueous Nb-based solution and subsequently annealed in (b) Ar and (c) NH_3 flows at 873 K for 1 h.

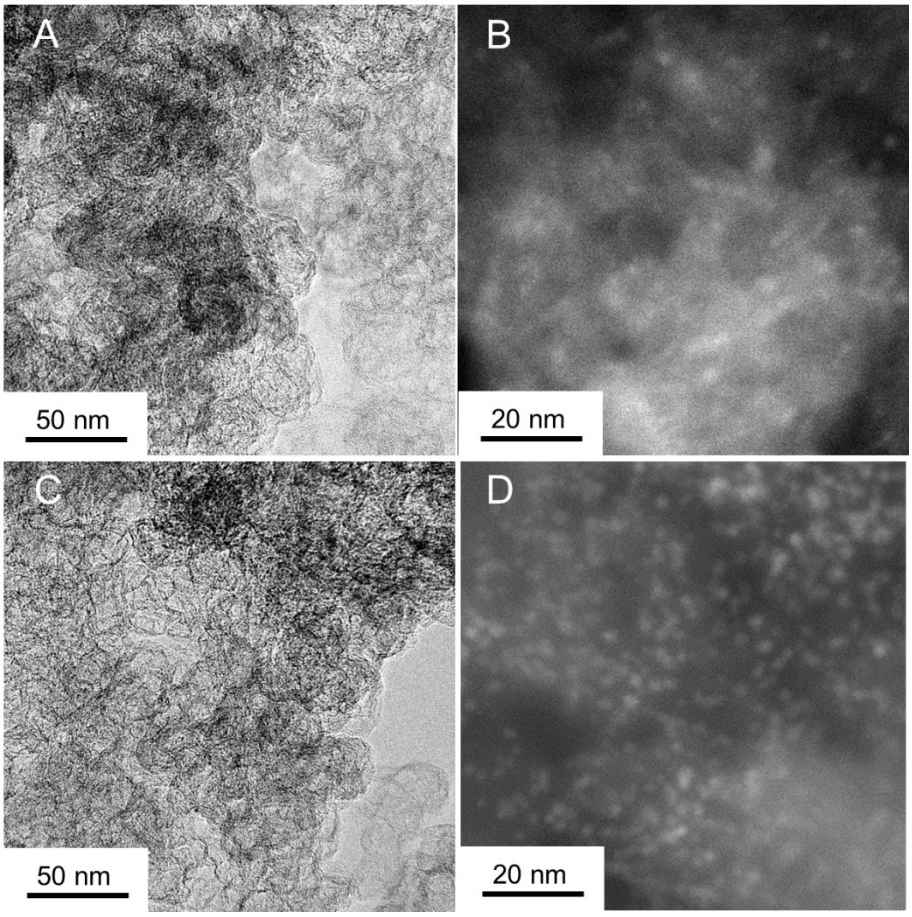

Figure S2. (A, C) TEM and (B, D) STEM images of the NbO_x/CB nanoparticles, (A, B) as-prepared by electrodeposition and (C, D) subsequent annealing treatment in an Ar flow at 873 K for 1 h.

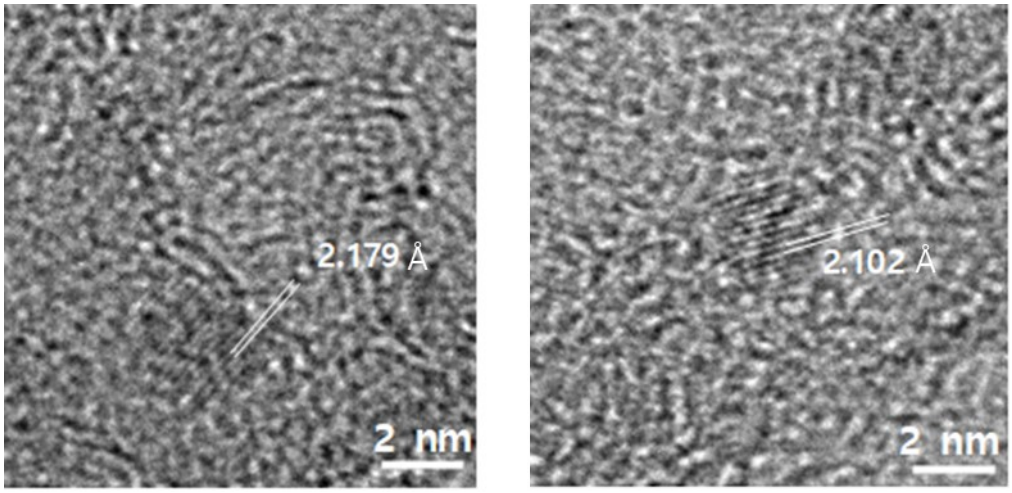

Figure S3. Magnified TEM images of the NbO_x nanoparticles shown in Figure 1D, prepared by electrodeposition and subsequent annealing treatment in NH_3 flow at 873 K for 1 h.

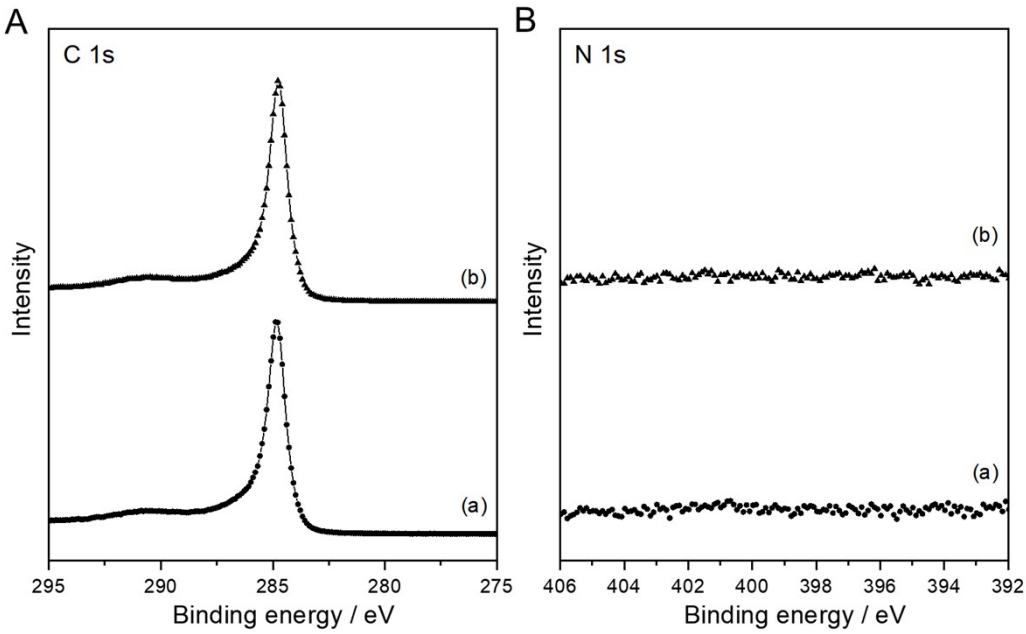

Figure S4. Narrow-scan (A) C 1s and (B) N 1s XPS spectra of CB particles (a) as-purchased and (b) annealed in NH_3 flow at 873 K for 1 h.

Table S1. Surface fractions of Nb species on NbO_x/CB nanoparticles (a) as-deposited and subsequently annealed in (b) Ar and (c) NH_3 flows at 873 K for 1 h, respectively, which were estimated based on the narrow-scan Nb 3d XPS spectra in Figure 2.

Condition	Fractions of surface Nb species	
	Nb^{5+}/Nb_{total}	Nb^{4+}/Nb_{total}
(a) as-deposited	0.74	0.26
(b) annealed in Ar	0.77	0.23
(d) annealed in NH_3	0.84	0.16

Table S2. Fitting results, namely, resistances and constant phase element (CPE) values, of Nyquist plots of the NbO_x/CB nanoparticles series annealed in (a) NH_3 and (b) Ar flows at 873 K for 1 h, respectively, and (c) as-deposited, shown in Figure 5(B). The equivalent circuit model for best-fits of Nyquist plots was presented in the inset of the Figure 5(B). The EIS measurements were performed in the O_2-purged 0.1 M KOH aqueous solutions at the applied potential of 0.72 V_{RHE}. The EIS data were acquired in the frequency range from 10^4 to 10^{-1} Hz at an AC amplitude of 10 mV.

	R_s [Ω]	CPE-P; n	CPE-T; Q [$\Omega^{-1}s^n$]	R_{ct} [Ω]
(a) annealed in NH_3	39.6	0.89	8.0×10^{-4}	241.0
(b) annealed in Ar	42.5	0.89	7.7×10^{-4}	321.6
(c) as-deposited	38.8	0.93	6.0×10^{-4}	812.2

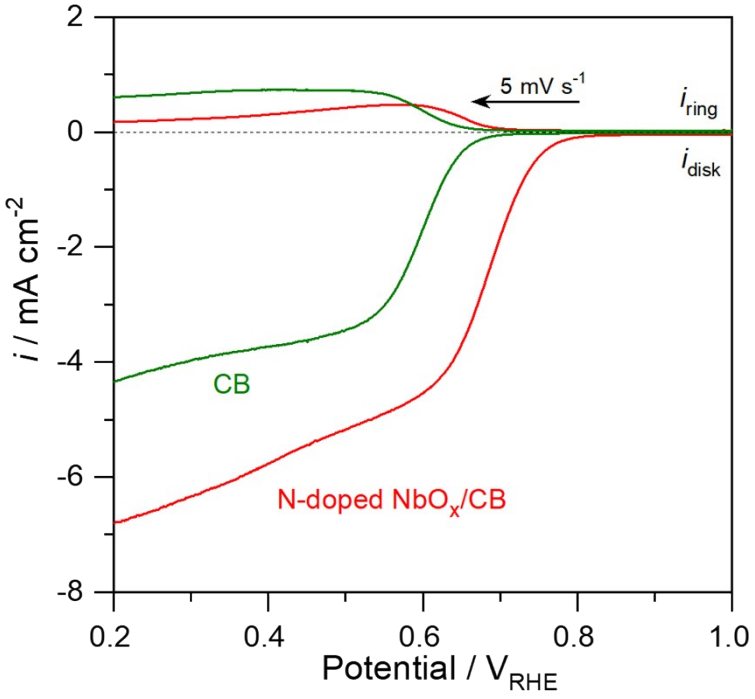

Figure S5. LSVs of the RRDE measurements for ORR over the prepared N-doped NbO_x/CB and CB catalysts in a O_2-purged 0.1 M KOH aqueous solution at a revolution rate of 1600 rpm and a scan rate of 5 mV s^{-1}. A constant potential of 1.2 V_{RHE} was applied to a Pt ring electrode. The H_2O_2 formation was estimated from the ring current.

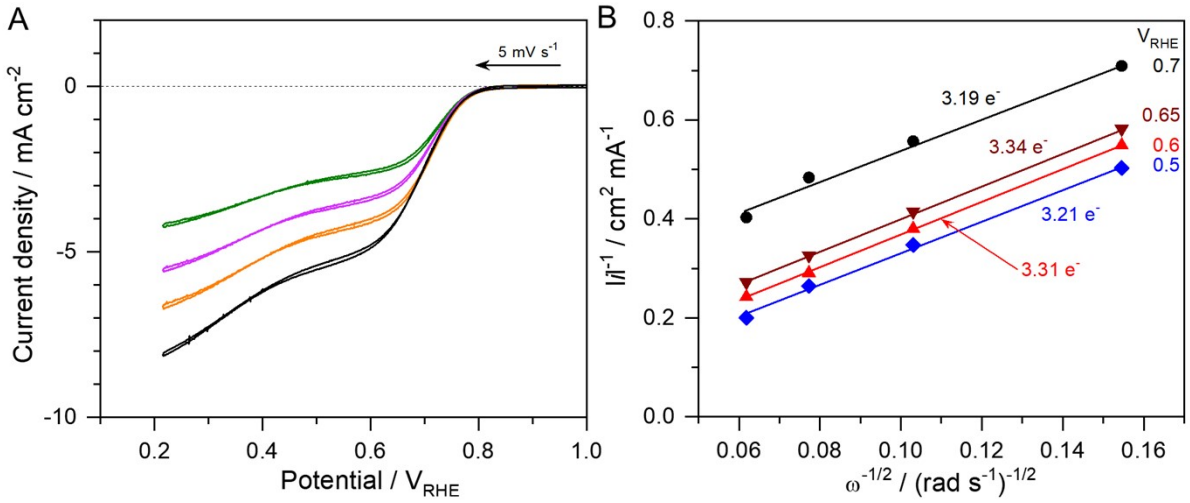

Figure S6. CVs of the prepared N-doped NbO_x/CB in a O_2-purged 0.1 M KOH aqueous solution at a scan rate of 5 mV s^{-1}, obtained by varying rotation speeds of 400, 900, 1600, and 2500 rpm. (B) Koutechky-Levich plots, $|i|^{-1}$ versus $\omega^{-1/2}$, of N-doped NbO_x/CB nanoparticles, which were calculated from the LSVs in (A). The electron transfer numbers derived from the slopes were provided for each potential.