Electronic Supplementary Material (ESI) for Journal of Materials Chemistry A.

Compact carbon nitride based copolymer film with controllable thickness for photoelectrochemical water splitting

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Preparation of g-C_3N_4/FTO photoelectrode

Typically, the FTO glass with a size of 1.0×2.5 cm was washed in turn with acetone, ethanol, and DI water under continuous sonication, and then dried in N_2 flowing. By dispersing a certain amount of g-C_3N_4 in water, the sample slurry was obtained and used for spreading onto the cleaned FTO glass substrate (photoactive area of 1.5 cm^2), and dried at room temperature. Uncoated areas on the electrode were isolated with insulating tape.

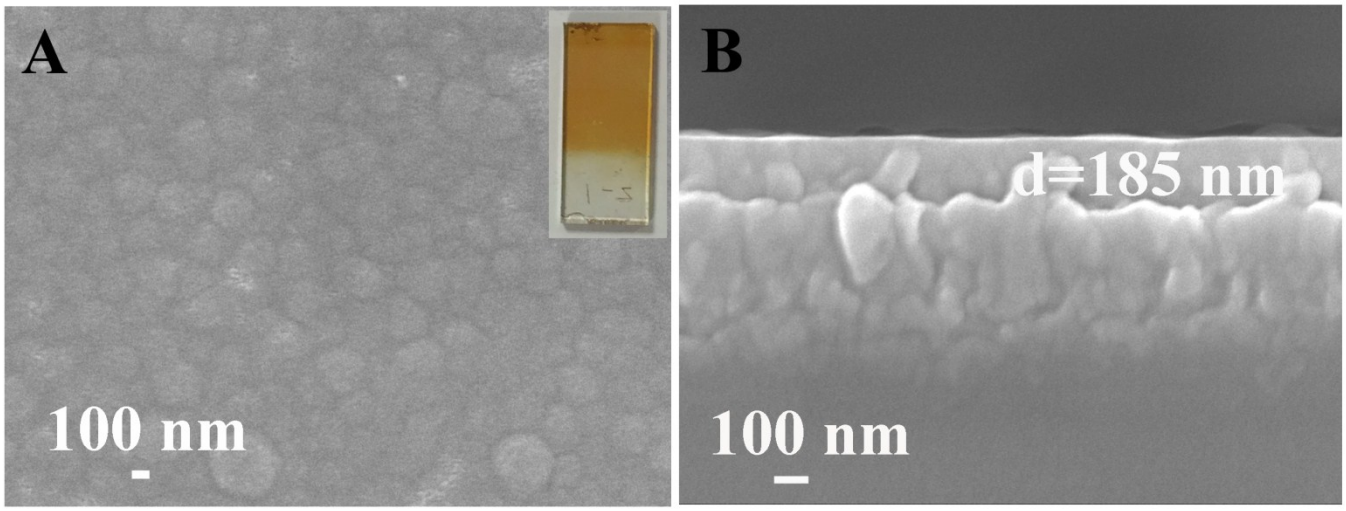

Figure S1 SEM images of top view (A) and cross section (B) of CNBC/FTO obtained at 400 °C annealing. Inset of A shows its digital photograph.

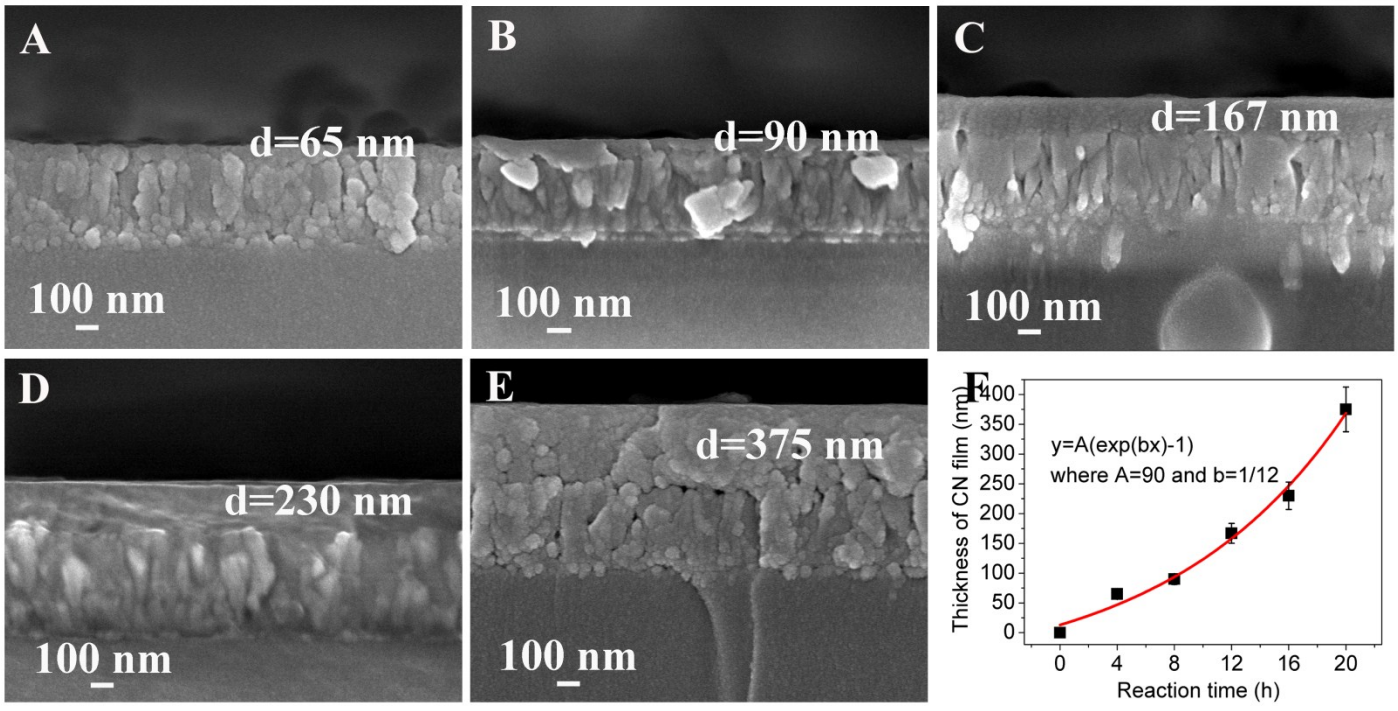

Figure S2 SEM images of cross section of as-prepared CNBC-U/FTO film obtained by different reaction time. (A) 4 h, (B) 8 h, (C) 12 h, (D) 16 h, (E) 20 h. (F) shows the relationship between reaction time and thickness of CNBC-U film

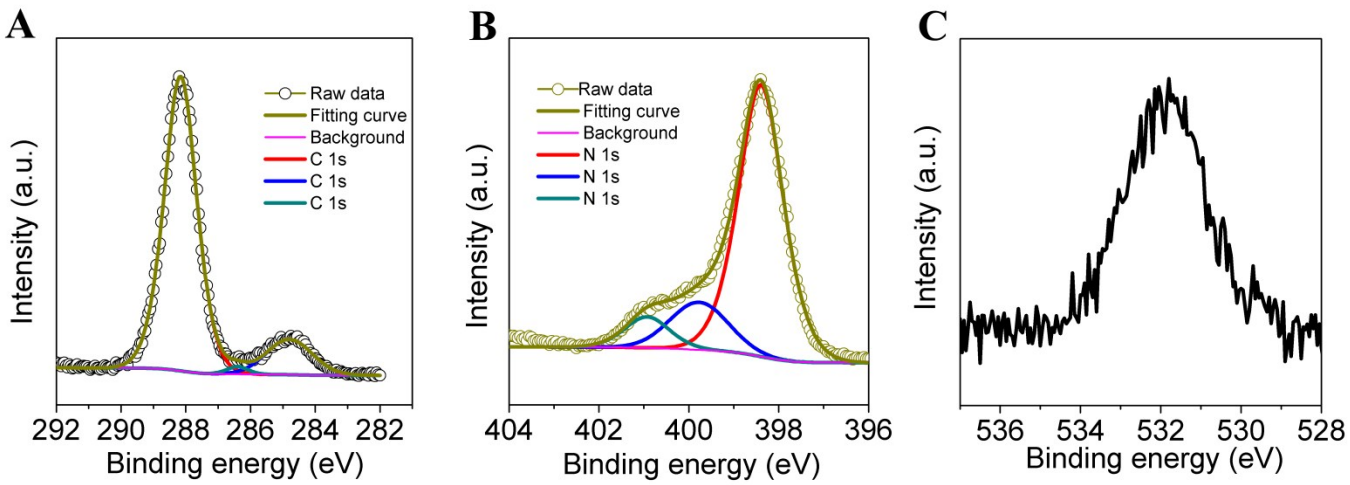

Figure S3 the high-resolution C 1s (A), N 1s (B), and O 1s (C) XPS spectra of g-C_3N_4/FTO.

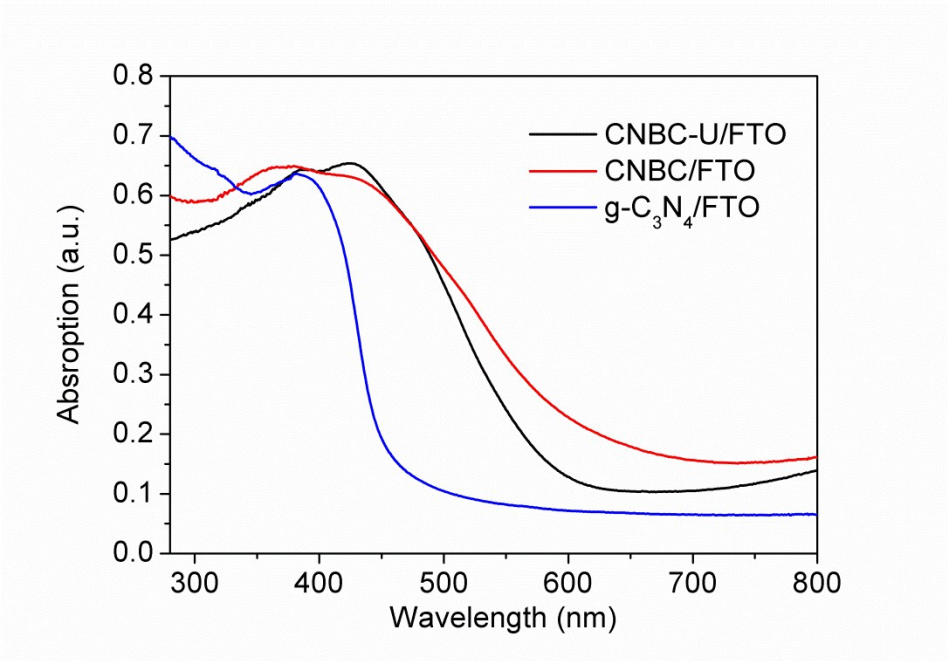

Figure S4 UV-visible absorption spectra of CNBC-U/FTO, CNBC/FTO and g-C_3N_4/FTO.

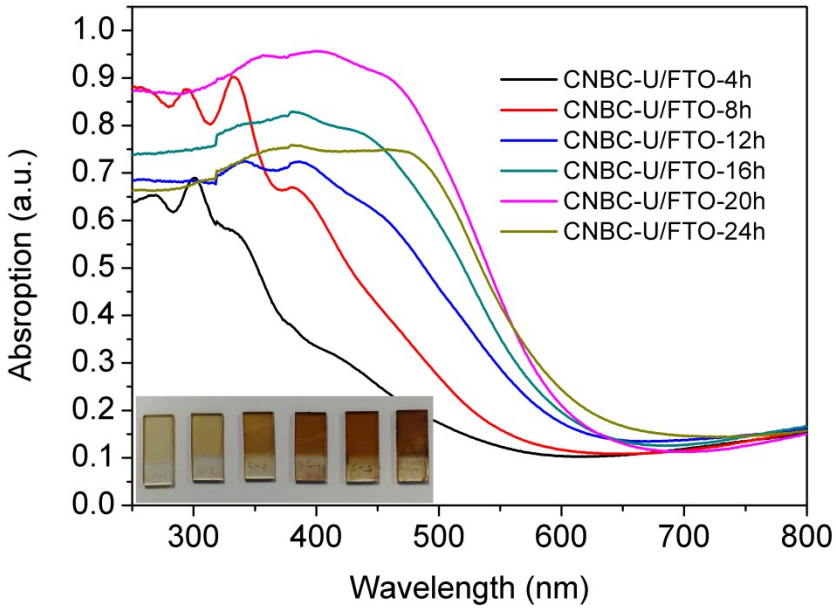

Figure S5 UV-Vis absorption spectra of as-prepared CNBC-U/FTO film obtained at different reaction time; inset shows the picture of CNBC-U/FTO film obtained after reaction time for 4 h, 8 h, 12 h, 16 h, 20 h, 24 h.

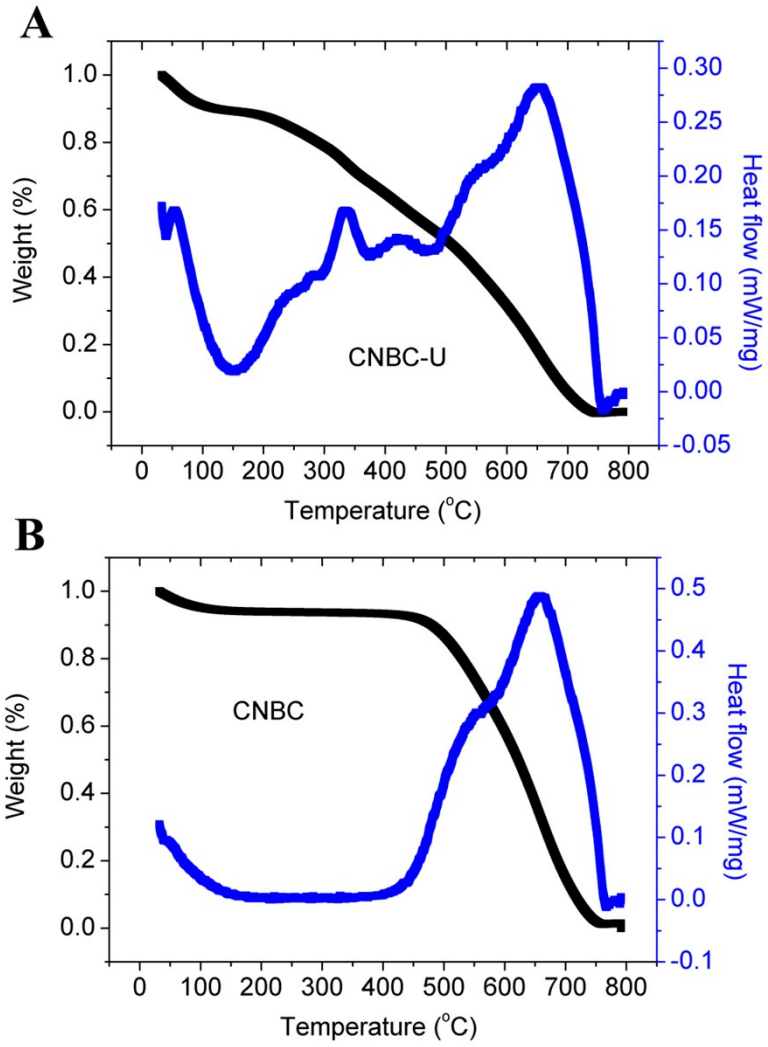

Figure S6 TGA and DSC curves of CNBC-U and CNBC powders measured in air and N_2.

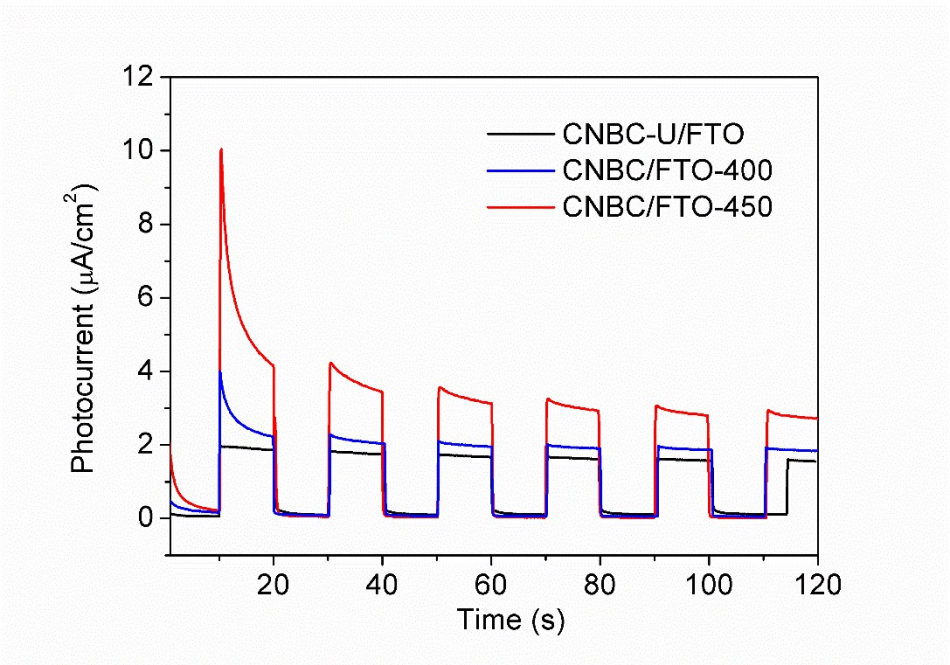

Figure S7 Photocurrent (i-t curve) of CNBC-U/FTO and CNBC/FTO annealed at different temperature (400 °C and 450 °C).

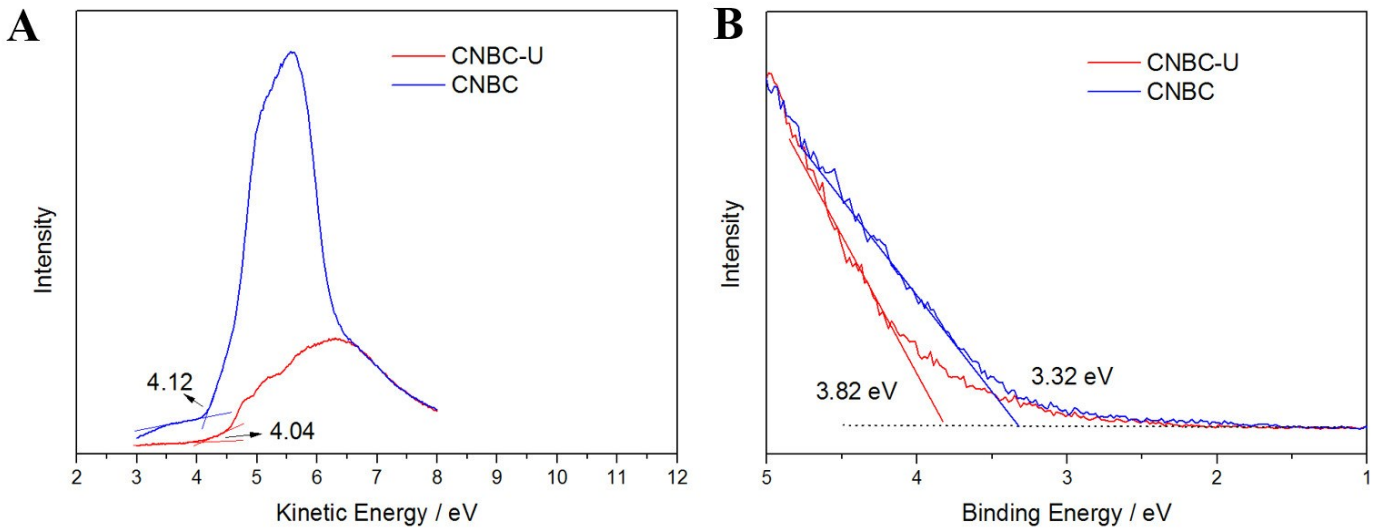

Figure S8 Results of Ultraviolet photoelectron spectroscopy (UPS) for CNBC-U and CNBC. The fitted values shown in (A) indicate the work function (WF) of the samples, and should be calibrated with 0.7 eV bias to a reference gold standard. The fitted values shown in (B) indicate the valence band (VB) with respect to Fermi level. Therefore, the estimated valence band levels for CNBC-U and CNBC are estimated as 2.72 V and 2.3 V (vs. SHE), respectively, according to the following table.

	WF (Figure S8A)	WF calibrated (with 0.7 eV bias)	VB-Fermi (Figure S8B)	VB vs. vacuum (WF calibrated + VB-Fermi)	VB vs. SHE (SHE as -4.44 eV against vacuum level)
CNBC-U	4.04	3.34	3.82	7.16	2.72
CNBC	4.12	3.42	3.32	6.74	2.3

Table S1. Element contents of C, N, O, and Cl determined by elemental analysis and XPS spectra.

Samples	Elemental content (wt%)							
	Elemental analysis				XPS			
	C	N	Cl	H	C	N	Cl	O
g-C_3N_4	34.8	58.2	-	2.0	31.8	60.9	0	7.3
CNBC-U	31.0	38.9	5.4	3.0	39.1	38.4	4.4	16.8
CNBC	40.6	51.8	0	2.4	42.6	50.8	0	6.6