

**Al-coordination polymers-derived nanoporous nitrogen-doped  
carbon microfibers as metal-free catalysts for oxygen  
electroreduction and acetalization reaction**

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## Supporting information

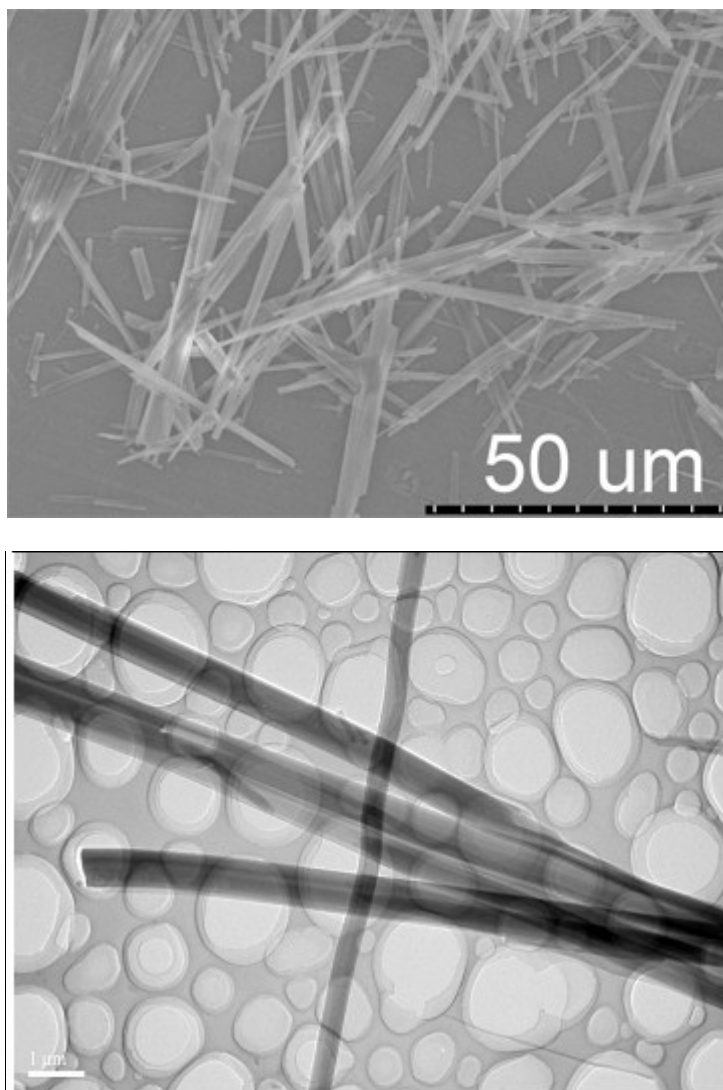


Figure S1 SEM and TEM images of Al-DTPA microfibers (80°C, DTPA and  $\text{Al}(\text{NO}_3)_3 \cdot 9\text{H}_2\text{O}$  is 1:1)

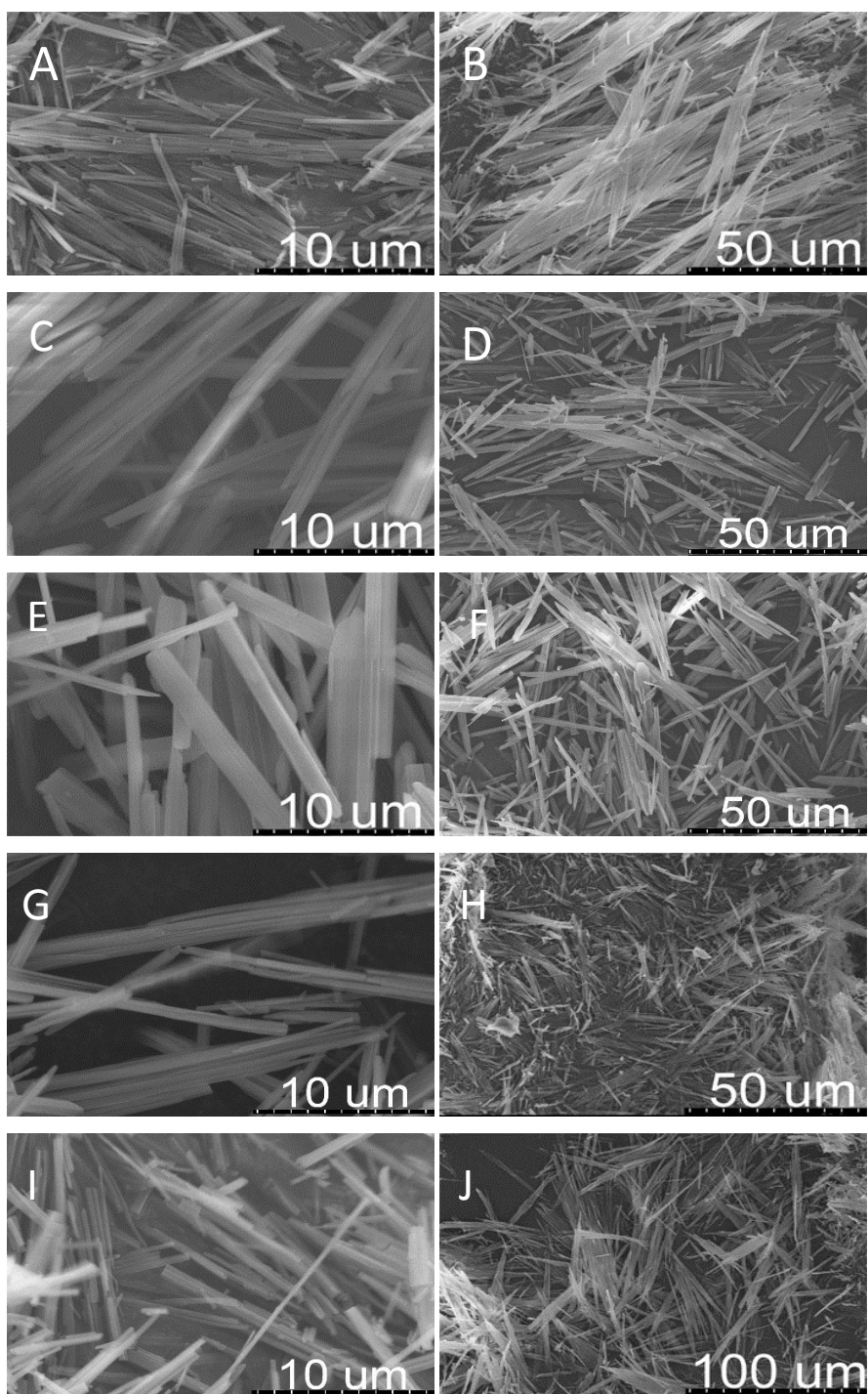


Figure S2 SEM images of Al-DTPA microfibers prepared at 60 (A and B), 80 (C and D) and 100 °C (E-F), respectively (the molar ratio of DTPA to  $\text{Al}(\text{NO}_3)_3 \cdot 9\text{H}_2\text{O}$  is 1:1); SEM images of Al-DTPA microfibers prepared at 60 °C with different molar ratios of DTPA to  $\text{Al}(\text{NO}_3)_3 \cdot 9\text{H}_2\text{O}$  ( 1:2, G and H; 2:1, I and J)

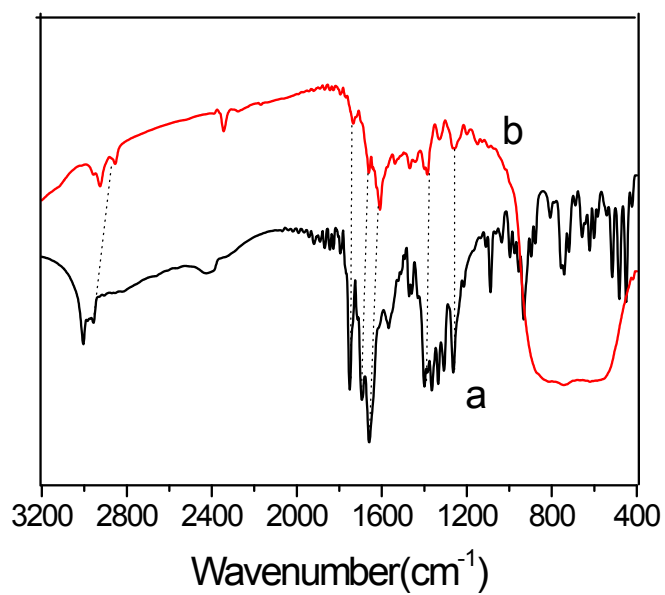


Figure S3 FT-IR spectra for DTPA (a) and Al-DTPA (b)

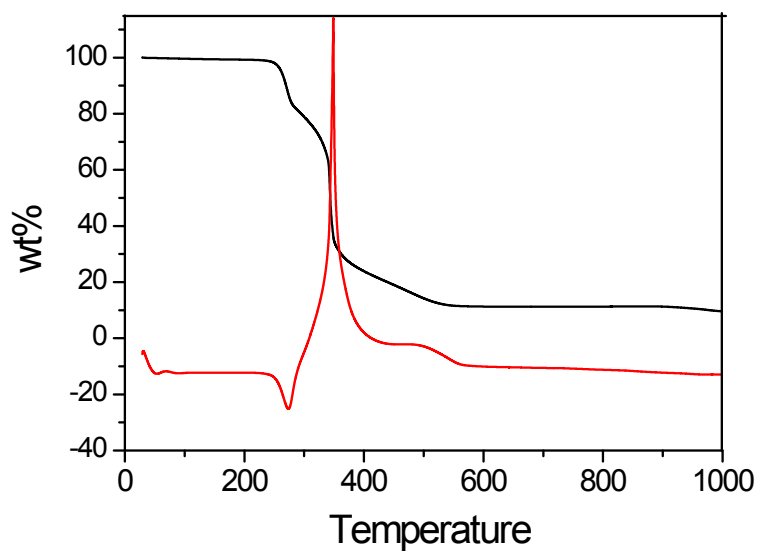


Figure S4 TG-DTA analysis for Al-DTPA microfiber in air

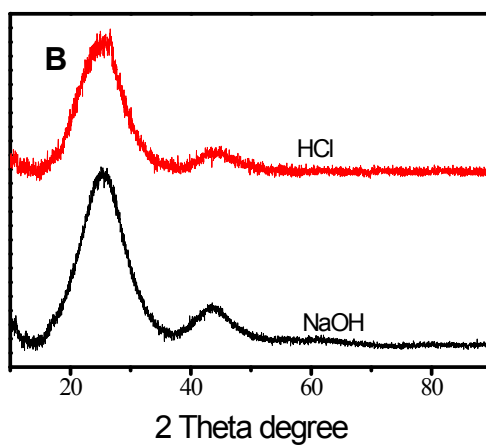
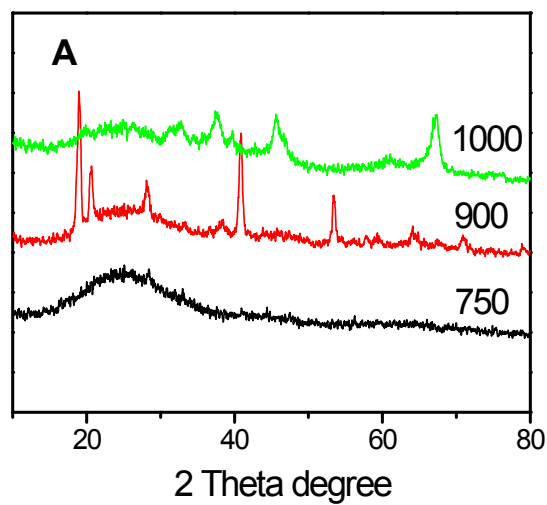


Figure S5 XRD patterns of the aluminium hydroxides or oxides-embedded carbon microfibers samples obtained by heating Al-DTPA at different temperatures (A). The NCF-900 obtained by etching off  $\text{Al}_2\text{O}_3$  or  $\text{Al}(\text{OH})_3$  materials with an aqueous solution of HCl and NaOH (10 wt%)(B).

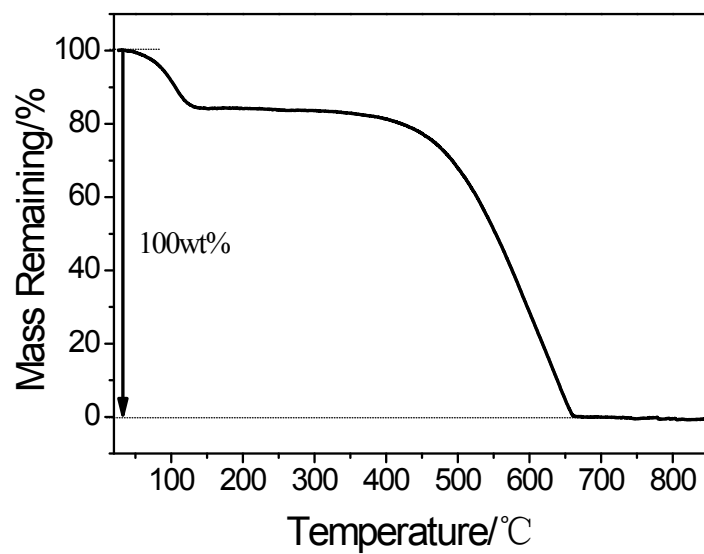


Figure S6 TGA analysis for NCF-900 in air

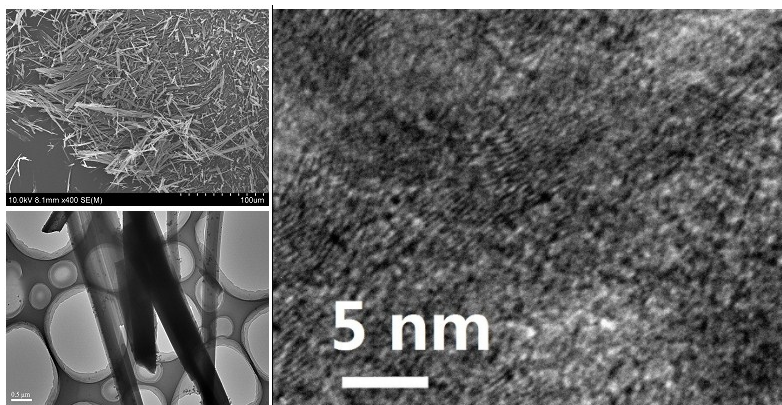


Figure S7 SEM, TEM and HRTEM images of NCFs obtained at 900 °C

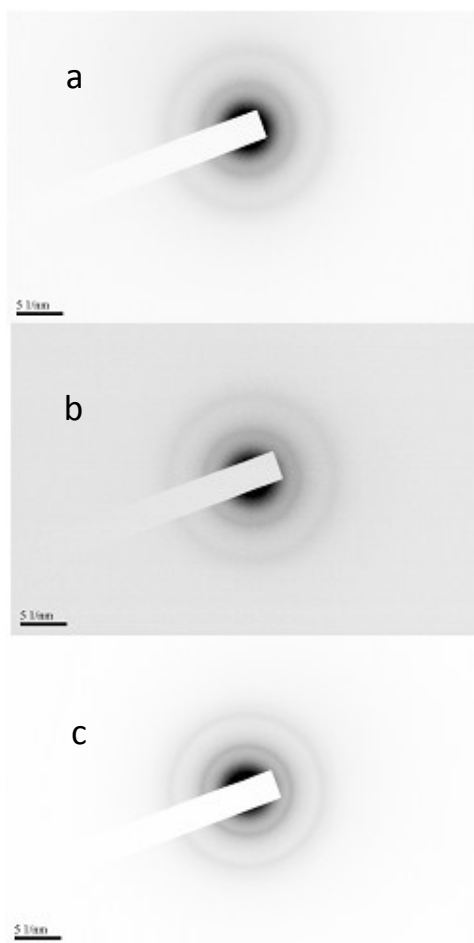


Figure S8 SAED images of NCFs obtained at (a) 750, (b) 900 and (c) 1000 °C in nitrogen atmosphere

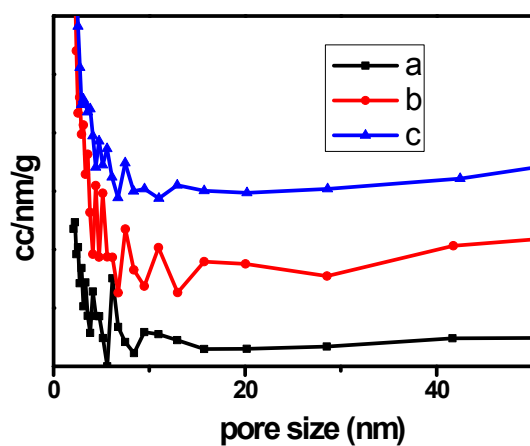


Figure S9 The pore size distribution curves for NCFs obtained at (a) 750, (b) 900 and (c) 1000 °C

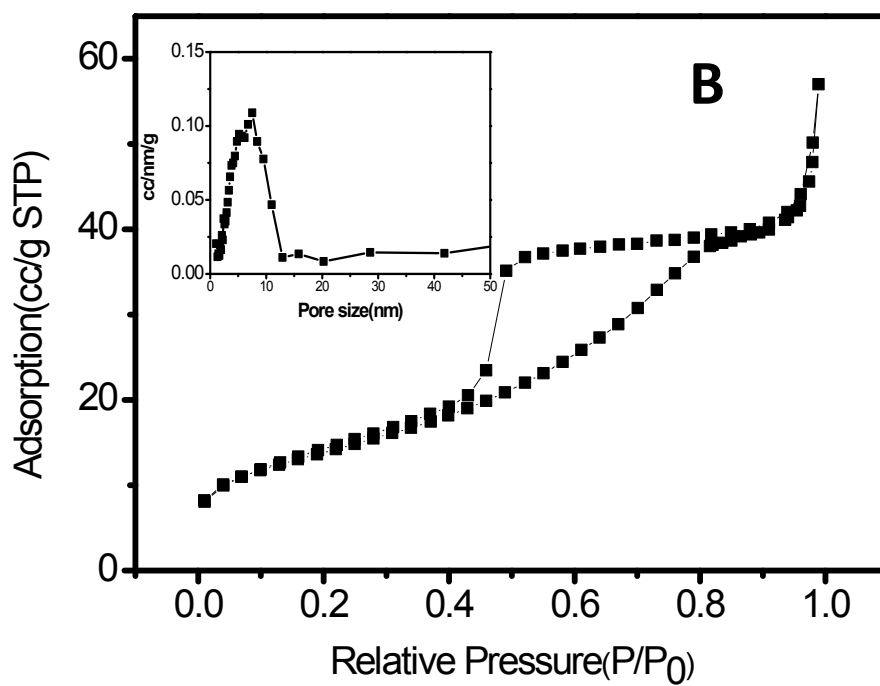
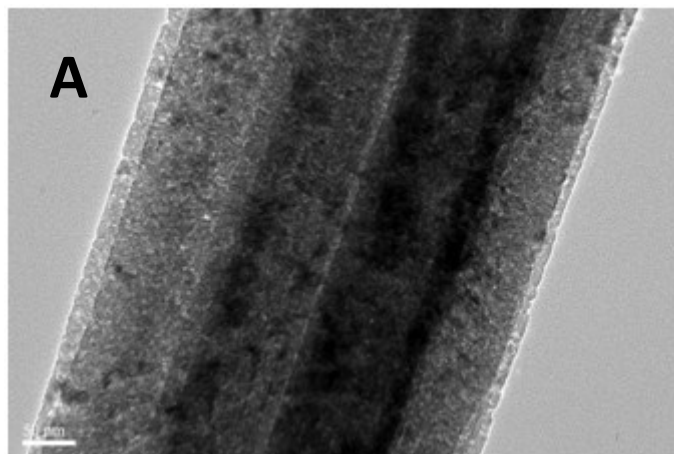


Figure S10 TEM (A) and N<sub>2</sub>-adsorption analysis (B) result for  $\gamma$ -Al<sub>2</sub>O<sub>3</sub> obtained by heating Al-DTPA at 800 °C in air



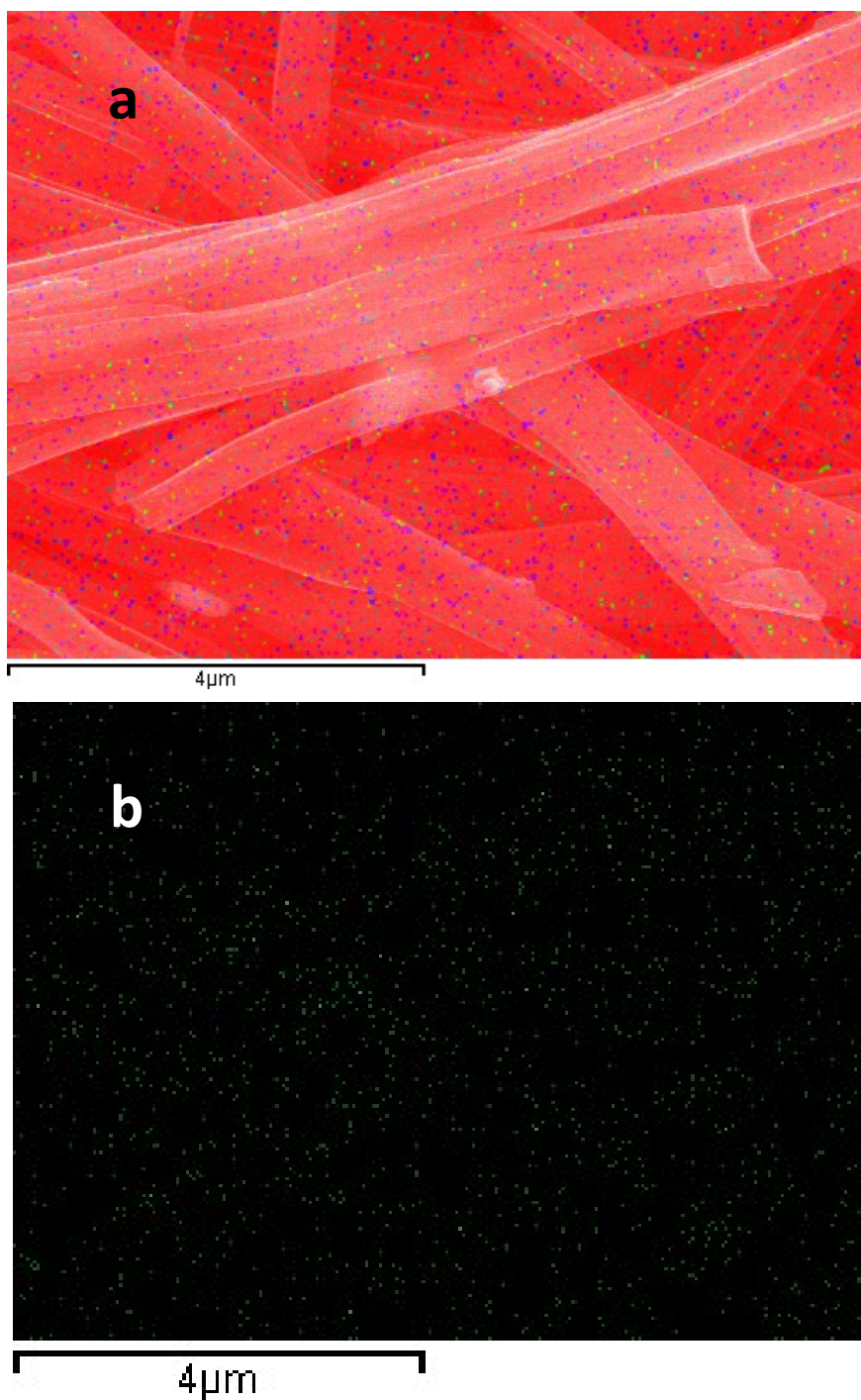


Figure S11 (a) SEM EDS element mapping images of NCF-900 for nitrogen, carbon and oxygen; (b) individual EDS element mapping image for nitrogen

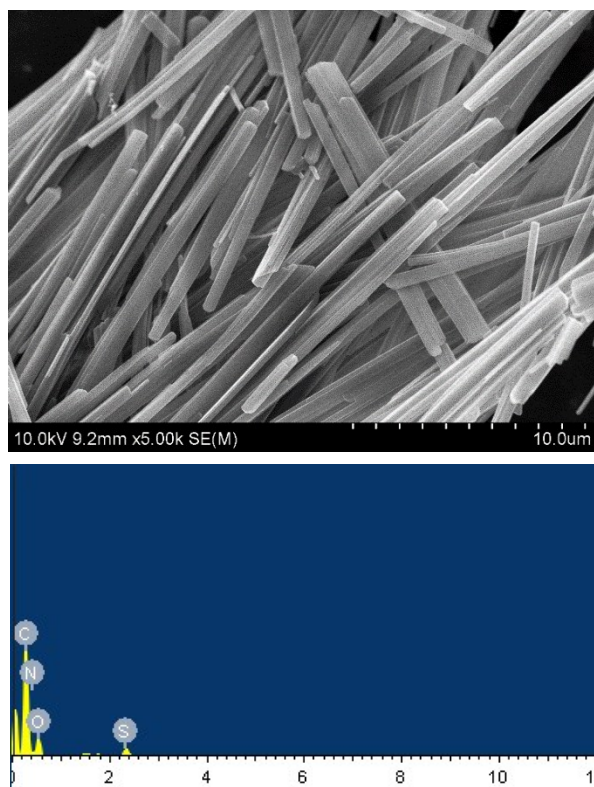


Figure S12 SEM and EDS spectrum of  $\text{SO}_3\text{H}/\text{NCF}-600$

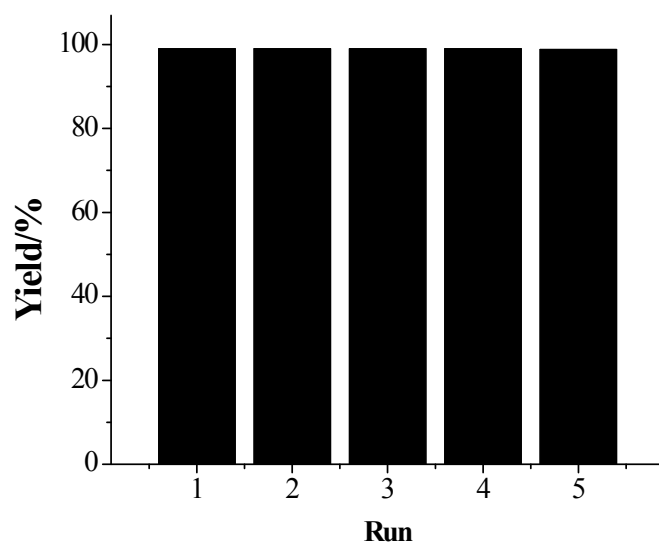


Fig.S13 The recycles of the  $\text{SO}_3\text{H}/\text{NCF}-600$  catalyst for the acetalization reaction of benzaldehyde with 1,2-ethanediol

Table S1 The surface species of N-doped carbon fibers and the content of deconvoluted N-type determined by XPS ( at %) and the nitrogen contents determined by elemental analysis ( wt %)

Samples	C (at%)	N (at %)	N <sup>a</sup> (wt%)	Py-N <sup>+</sup> -O <sup>-</sup> (%)	Py-like (%)	G-like (%)	Pyr-N-H (%)
750	83.1	7.9	8.5	9.7	50.8	11.1	28.4
900	83.7	6.7	7.0	7.8	52.2	21.5	18.6
1000	84.0	3.6	3.9	12.0	34.9	50.0	3.2

<sup>a</sup> determined by elemental analysis

Table S2 The surface species and nitrogen content of deconvoluted N-type determined by XPS for Al-DTPA -600 and SO<sub>3</sub>H/NCF-600

Samples	C (at %)	N (at %)	Py-N <sup>+</sup> -O <sup>-</sup> (%)	Py-like (%)	G-like (%)	Pyr-N-H (%)
Al-DTPA -600	51.08	4.84	10.1	51.3	10.4	28.2
SO <sub>3</sub> H/NCF-600	68.40	11.03	12.6	21.6	25.8	40.0