Electronic Supplementary Material (ESI) for New Journal of Chemistry.

Experimental Section

1.1 Materials

Melamine, ethanol, triethanolamine were directly used without further treatments.

1.2 Synthesis of PCN

Polymeric carbon nitride (PCN) was prepared via traditional thermal polymerization method.[1] 10 g melamine were heated in a muffle furnace at 550 °C for 4 h with a rate of 10 °C/min. Light yellow catalysts were collected and used without further treatments after cooled down to the room temperature.

1.3 Synthesis of PCN-SR

PCN-SR was prepared by LiOH post-heat treatment. 0.5 g PCN were mixed with different amounts of LiOH (40 mg, 80 mg, 120 mg and 160 mg) in vacuum glove box. Then, the mixed powders were heated to 500 °C at a rate of 15 °C/min in a muffle furnace and kept for 2 h. After cooled to the room temperature, the deep yellow solid powders were washed with hot deionized water for several times. After that, the wet powders were dried in a vacuum oven. The obtained catalyst was named as PCN-SRx (x= 40, 80, 120, 160), where x represented the amount of LiOH.

1.4 Characterization

X-ray diffraction (XRD) and Fourier transform infrared spectroscopy

(FT-IR) were conducted by Bruker D8 Advance and Thermo Nicolet IS 50, respectively. UV-Vis diffuse reflectance spectroscopy was conducted with Lambda 650s. The surface structures of samples were conducted with X-ray photoelectronic spectrum (Thermo ESCALAB 250) with a monochromated Al Kα and used 284.8 eV as a calibration. Transmission electron microscopy (TEM) was performed by FEI Tecnai 12. Time solved fluorescence spectra (TR-PL) was conducted by Edinburgh FLS1000. Photoelectronic tests were conducted with CHI 660 based on standard three-electrodes system with Pt foil as counter electrode and Ag/AgCl electrode as reference electrode.

1.5 Photocatalytic activity

Photocatalytic H_2O_2 reaction was evaluated under 420 nm LED irradiation with self-made reactor. The reaction was carried out under a mixed solvent consisted of water and methanol (27 ml:3 mL). 50 mg catalysts were dispersed into the solvent. After freezing with liquid N_2, the reaction system was ventilated with oxygen and used O_2 (~0.1 MPa) for protection. With 420 nm LED as light source, the reaction was carried out at room temperature for 1 h. H_2O_2 concentration was analyzed by measuring the absorbance intensity of the Ti^{IV}-H_2O_2 yellow complex at 410 nm with UV–Vis spectra.[2]

The apparent quantum yield (AQY) was calculated as followed:

$$AQY = \frac{N_e}{N_p} \times 100\% = \frac{2MhcN_A}{SPt\lambda} \times 100\%$$

Where the N_e, N_p, M, h, c, N_A, S, P, t and λ present the amount of reaction photons, incident photons, the amount of H_2O_2 molecules, the Plank constant, the light speed, Avogadro's constant, the irradiation area, the irradiation intensity, the light wavelength, respectively.

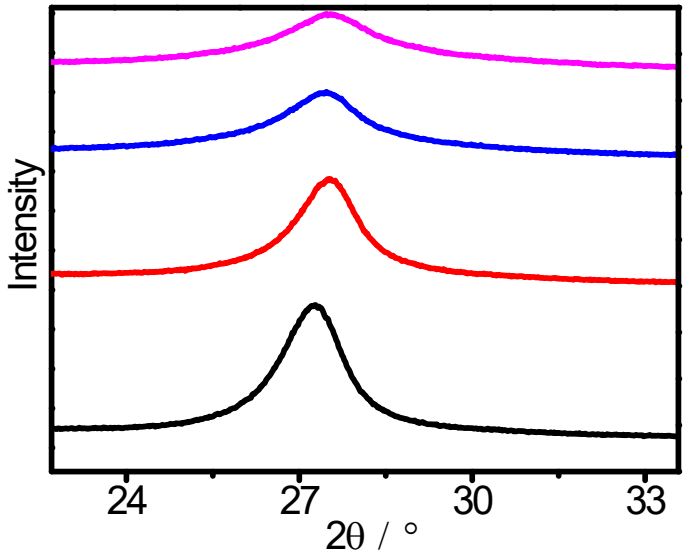

Fig. S1 the enlarged XRD patterns of samples

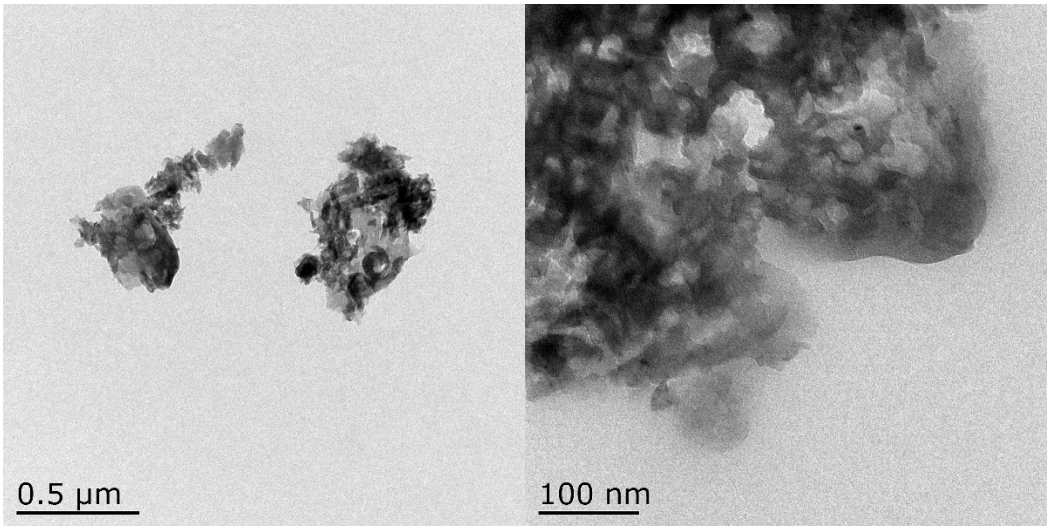

Fig. S2 TEM images of PCN

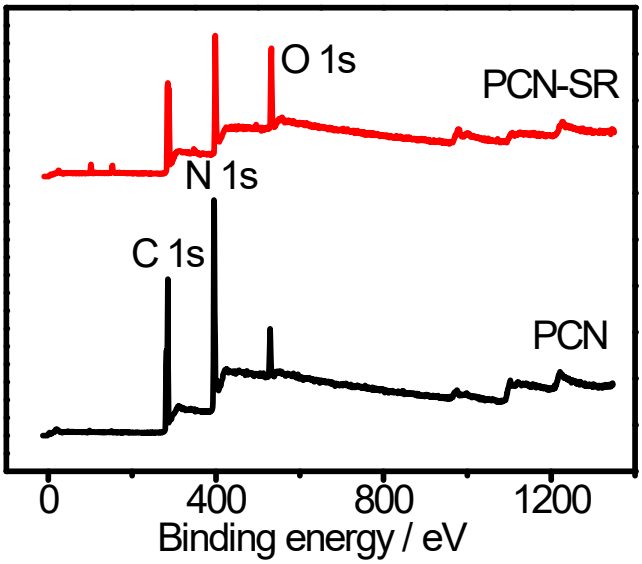

Fig. S3 XPS survey spectra of samples

Table S1 The element contents of C, N, Li of PCN and PCN-SR

Samples	atomic ratios				
	C %	N %	O %	Li %	C/N
PCN	24.93	21.26	2.91	0	1.17
PCN-SR	26.92	14.90	6.52	1.67	1.81

Table S2 The relative content of C-N=C, C-N_3 and C-NH_x

Samples	relative content		
	C-N=C	C-N3	C-NHx
PCN	78.5	16.0	5.5
PCN-SR	76.2	17.8	6.1

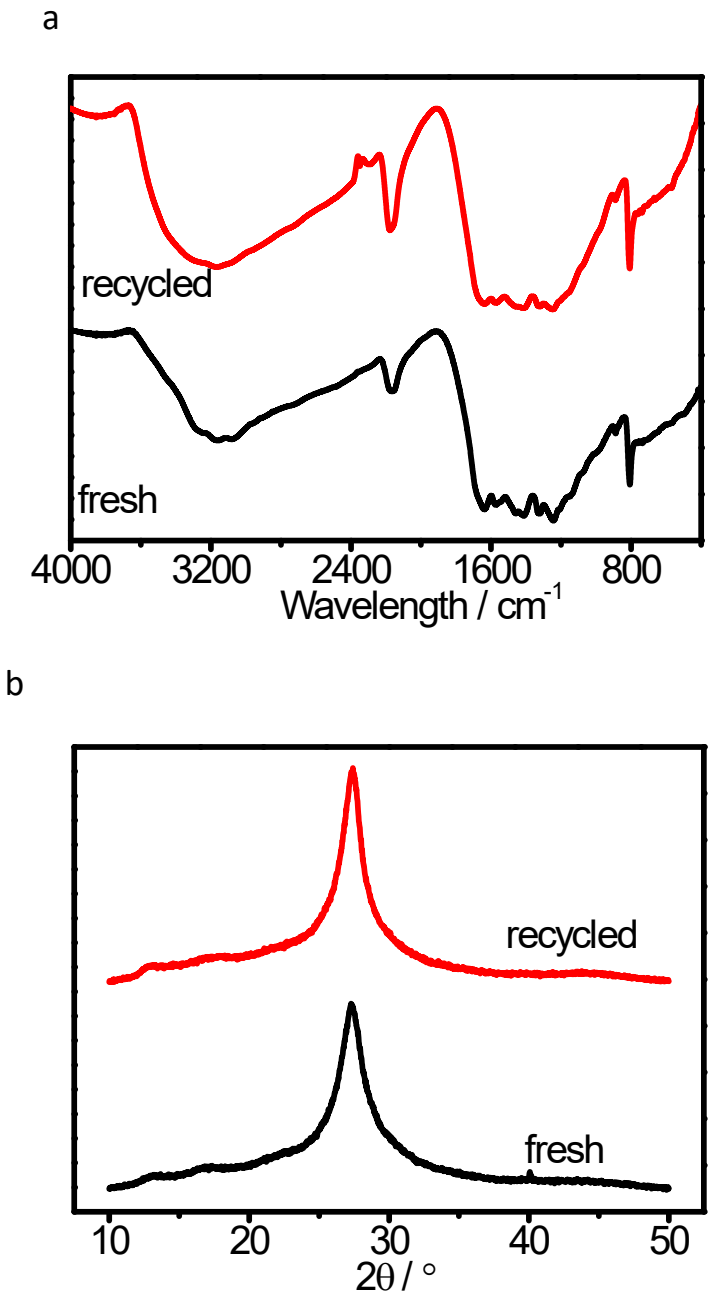

Fig. S4 (a) FT-IR spectra and (b) XRD patterns of recycled samples

1. X. Wang, K. Maeda, A. Thomas, K. Takanabe, G. Xin, J. M. Carlsson, K. Domen and M. Antonietti, *Nat. Mater.*, 2009, **8**, 76-80.
2. H. Ou, C. Tang, X. Chen, M. Zhou and X. Wang, *ACS Catal.*, 2019, 2949-2955.