

Supporting Information for:

**Gas-Thermal Method as a Universal and Convenient Strategy for Preparing  
Non-traditional Luminescent Polymers with Enhanced and Red-Shifted  
Fluorescence**

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## EXPERIMENTAL SECTION

**Materials.** Poly(vinyl alcohol) (PVA, 98% purity,  $M_w=7.7\times 10^4$ ) was purchased from Sinopharm Chemical Reagent Co., Ltd. High-purity nitrogen was purchased from Beijing Huanyu Jinghui Jingcheng Gas Technology Co., Ltd. Poly(ethylene glycol) (PEG,  $M_w=2\times 10^4$ ) was purchased from Beijing Solarbio Science & Technology Co., Ltd. Polyamide-66 (PA66) was purchased from Shanghai Adamas Reagent Co., Ltd. Formic acid (98.0% purity) was purchased from Tianjin Kemiou Chemical Reagent Co., Ltd. Starch from potato was purchased from Aladdin Reagent Co., Ltd.

**Gas-thermal method.** 1-2 g sample was placed at the bottom of a quartz boat, which was settled in the middle of an NBD-0 tubular high temperature sintering furnace (Nobody Materials Science and Technology Co., Ltd., China), and then heated in air or nitrogen at different temperatures for different time periods. Nitrogen was pumped in for 10 minutes to remove the air before use. And it was pumped in slowly and continuously during the heating process.

**Fluorescence spectroscopy.** Fluorescence spectra as well as lifetimes were measured with an FLS-980 fluorescence spectrometer (Edinburgh instruments, UK) at room temperature. The excitation and the emission slit widths for the powders were 3 nm, if not otherwise stated. The luminescence quantum yields were measured with a Quantaaurus-QY absolute photoluminescence quantum yield spectrometer (Hamamtsu, Japan) at room temperature. Photographs of the solid powders under UV illumination and day light were taken with a Canon D600 camera in a dark room. The exposure time was automatically adjusted by the camera. The microscopic photos of product powders under the irradiation of lights with different excitation wavelengths were taken with an Olympus IX71 fluorescence microscope.

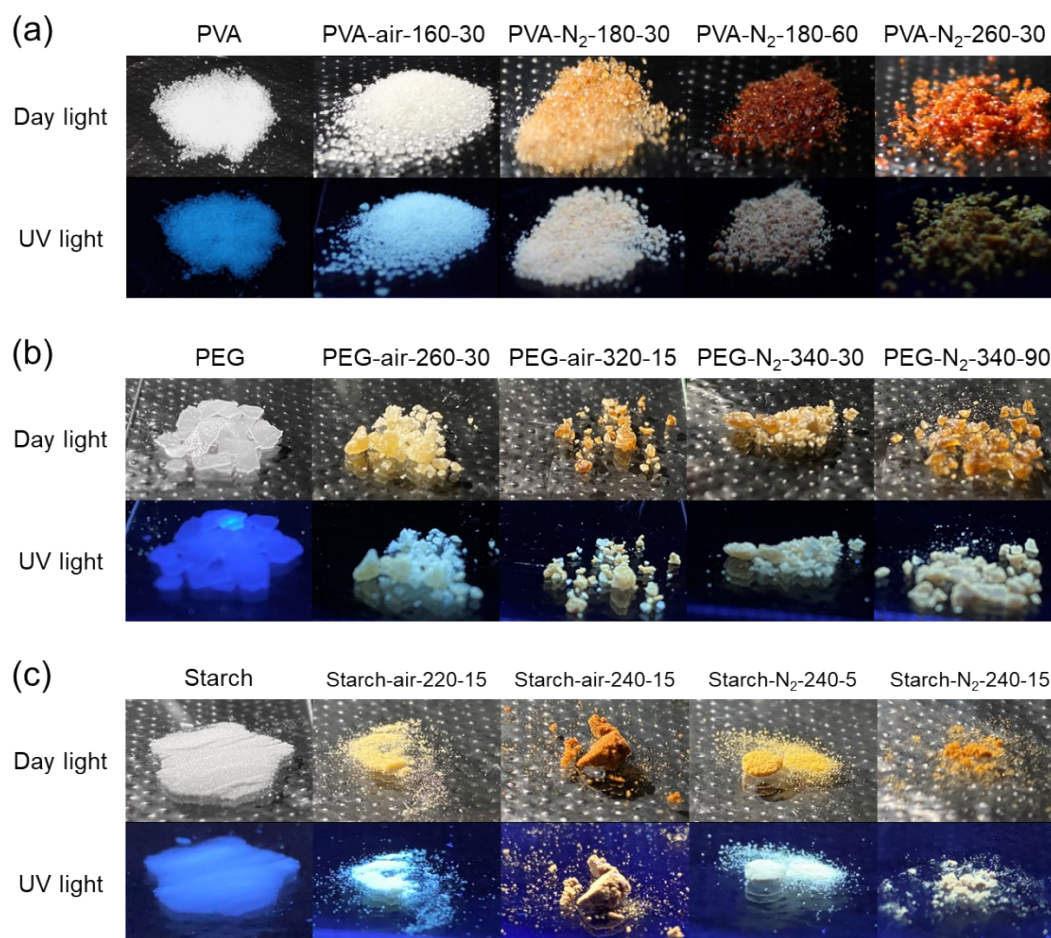
**UV-Vis spectroscopy.** UV-Visible (UV-Vis) spectra of the solutions were measured with a UV-2600 UV-Vis spectrophotometer (Shimadzu, Japan) at room temperature.

**FTIR-ATR characterization.** Fourier transform infrared (FTIR) spectra of the samples were recorded with a NEXUS 670 FTIR spectrometer (Thermo-Nicolet, USA). The samples were frozen and ground into powders in liquid nitrogen, and then subjected to attenuated total reflection (ATR) characterization with the sample surface in contact with diamond crystals at an incidence angle of 45°.

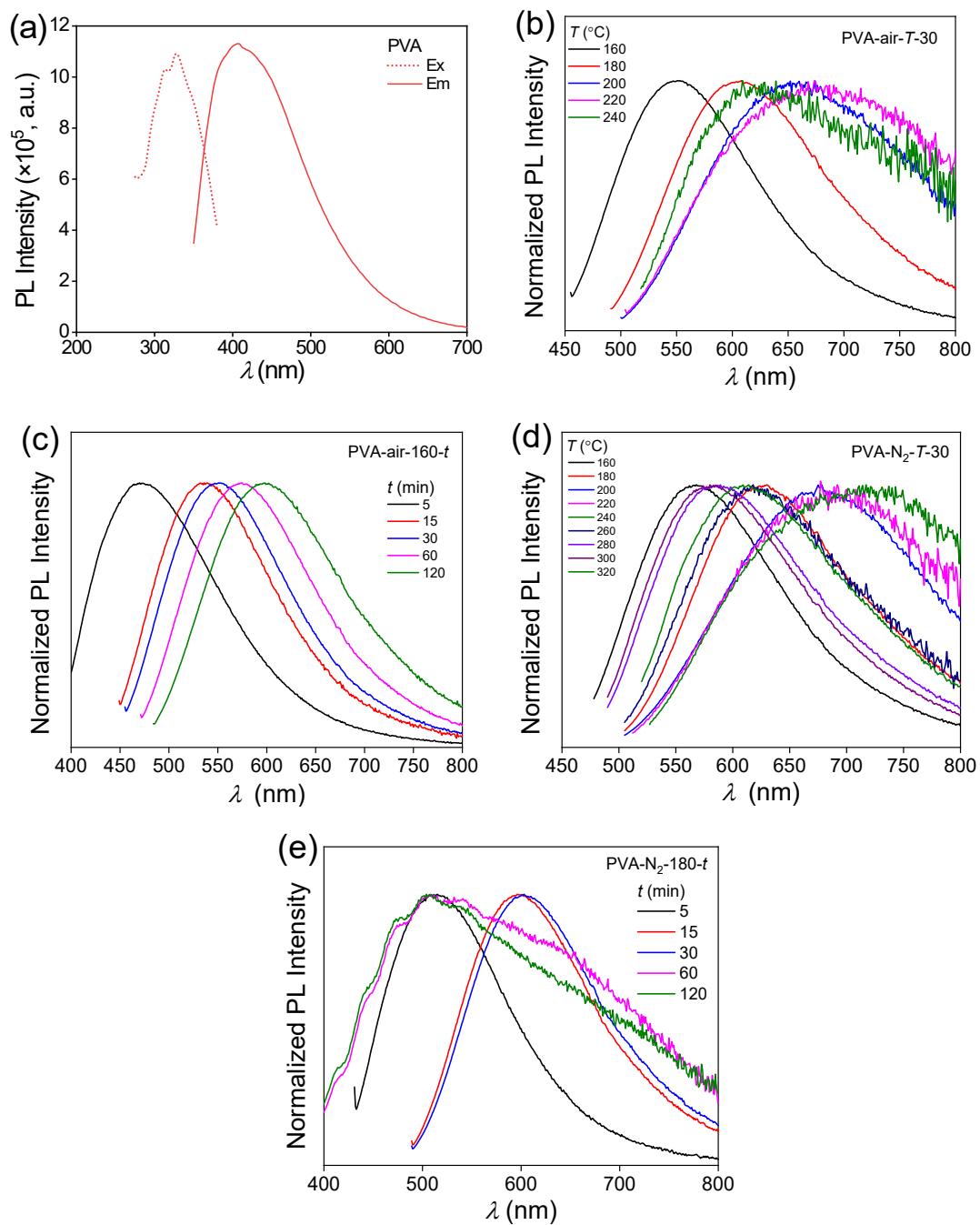
**XPS characterization.** X-ray photoelectron spectroscopy (XPS) spectra of the samples were recorded with a ESCSLAB250Xi electron spectrometer (Thermofisher, UK).

**TG analysis.** The thermogravimetric analysis (TGA) curves were recorded with an STA 449 F5 simultaneous thermal analyzer (Netzsch, Germany).

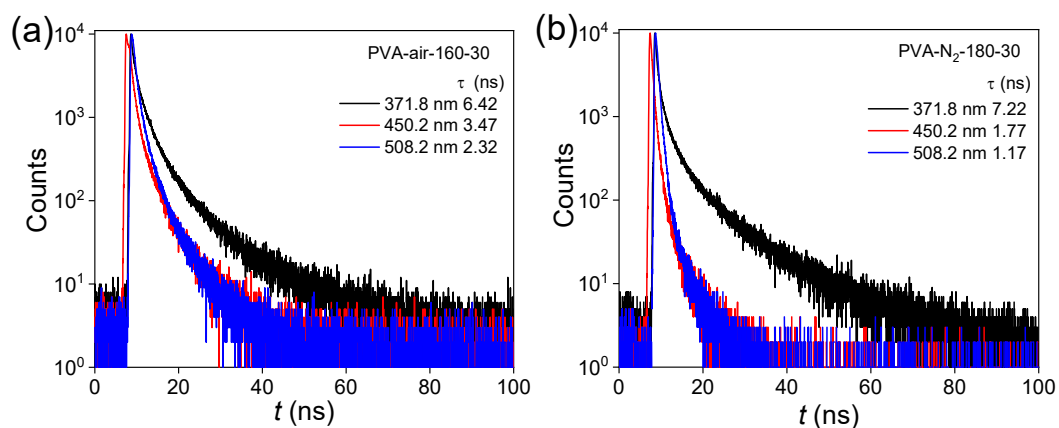
## SUPPORTING FIGURES AND TABLES



**Fig. S1** Photographs of PVA, PEG, starch and their gas-thermal products under the irradiation of day light and 365 nm UV light.



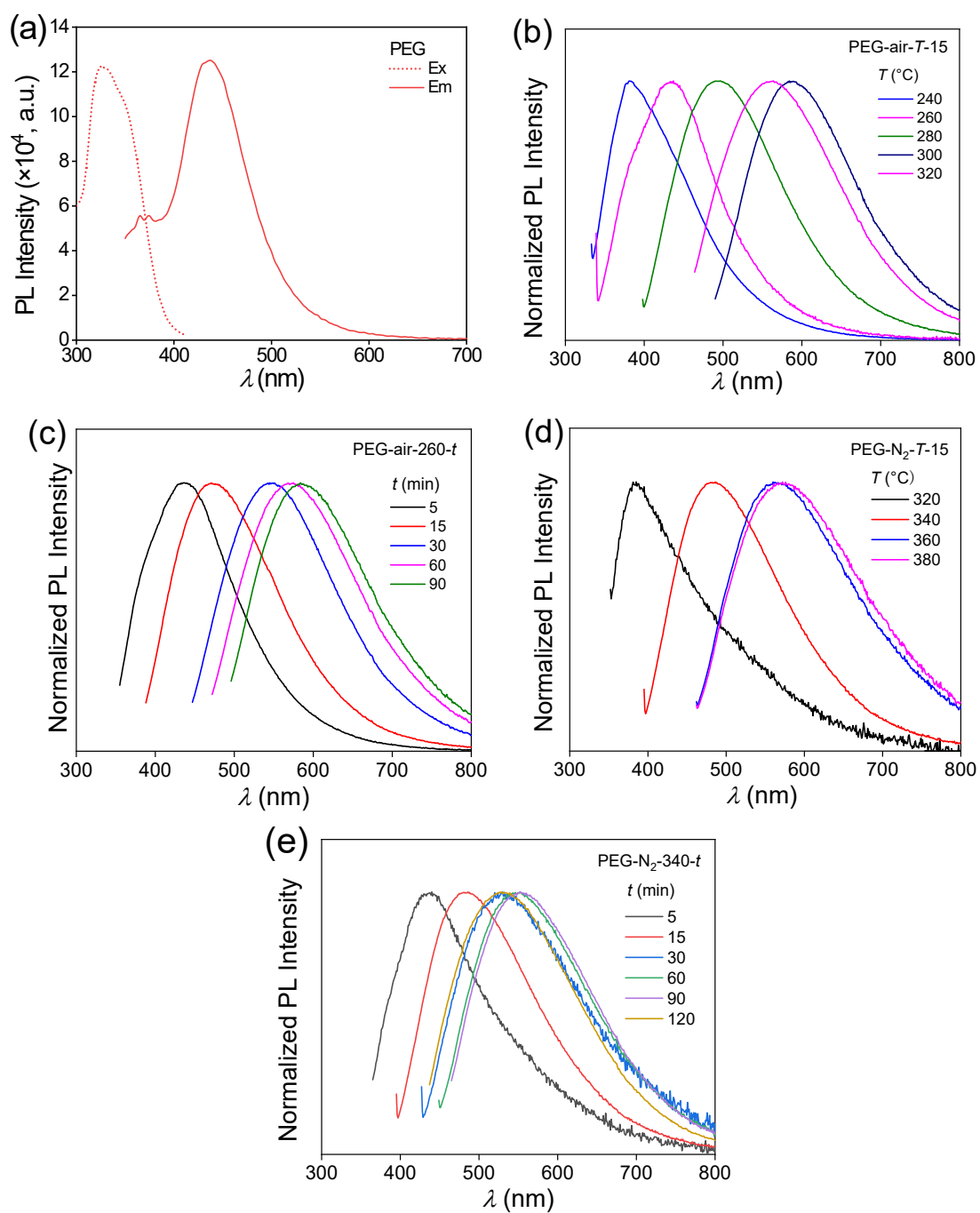
**Fig. S2** Fluorescence spectra of original PVA (a) and its gas-thermal product solids obtained under different conditions (b-e).



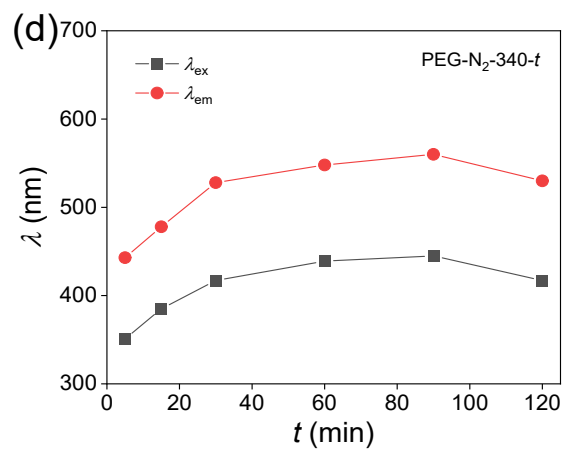
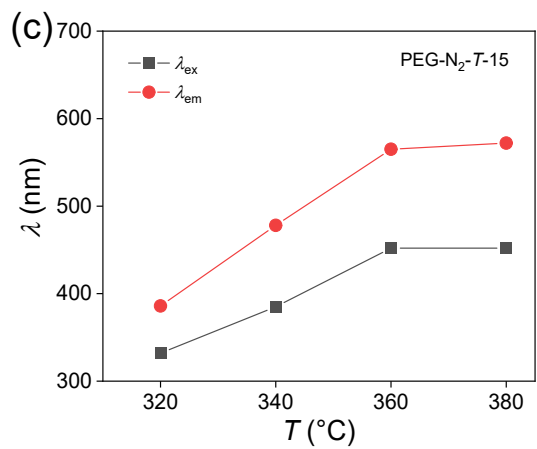
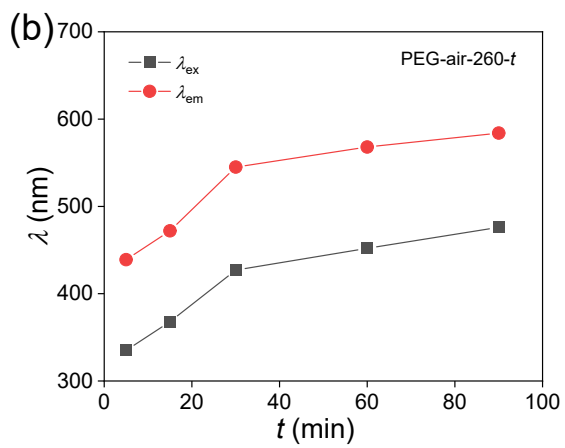
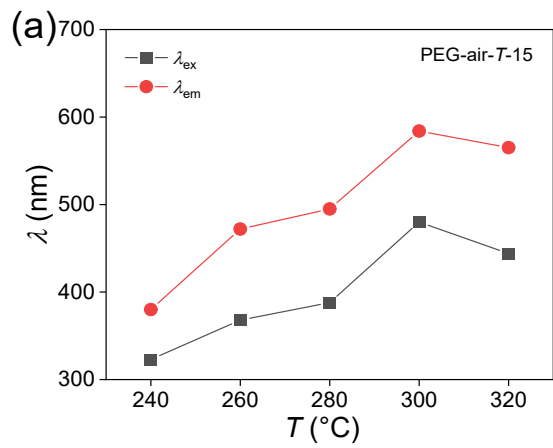
**Fig. S3** The fluorescence lifetimes of PVA gas-thermal product solids PVA-air-160-30 (a) and PVA-N<sub>2</sub>-180-30 (b) under different excitation wavelengths.

**Table S1** The quantum yields of PVA gas-thermal product solids.

Sample	QY (%)
PVA-N <sub>2</sub> -180-30	4.5
PVA-N <sub>2</sub> -220-30	0.6
PVA-N <sub>2</sub> -260-30	1.5
PVA-N <sub>2</sub> -300-30	1.9
PVA-N <sub>2</sub> -180-5	6.4
PVA-N <sub>2</sub> -180-120	1.0
PVA-N <sub>2</sub> -260-5	1.2
PVA-N <sub>2</sub> -260-120	1.3
PVA-air-160-30	6.0
PVA-air-200-30	1.0
PVA-air-240-30	0.2
PVA-air-160-5	1.8
PVA-air-160-120	3.3

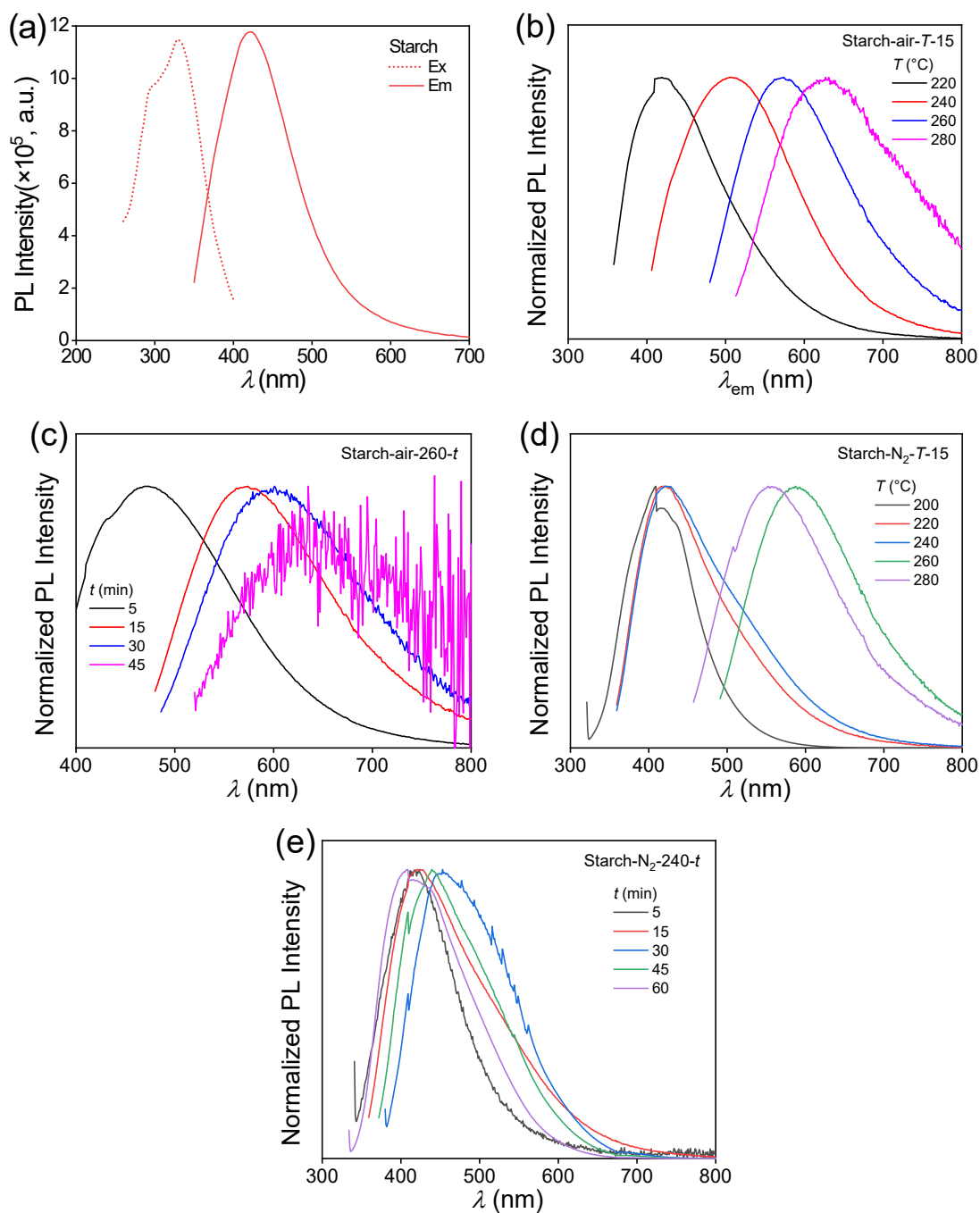


**Fig. S4** Fluorescence spectra of original PEG (a) and its gas-thermal product solids obtained under different conditions (b-e).

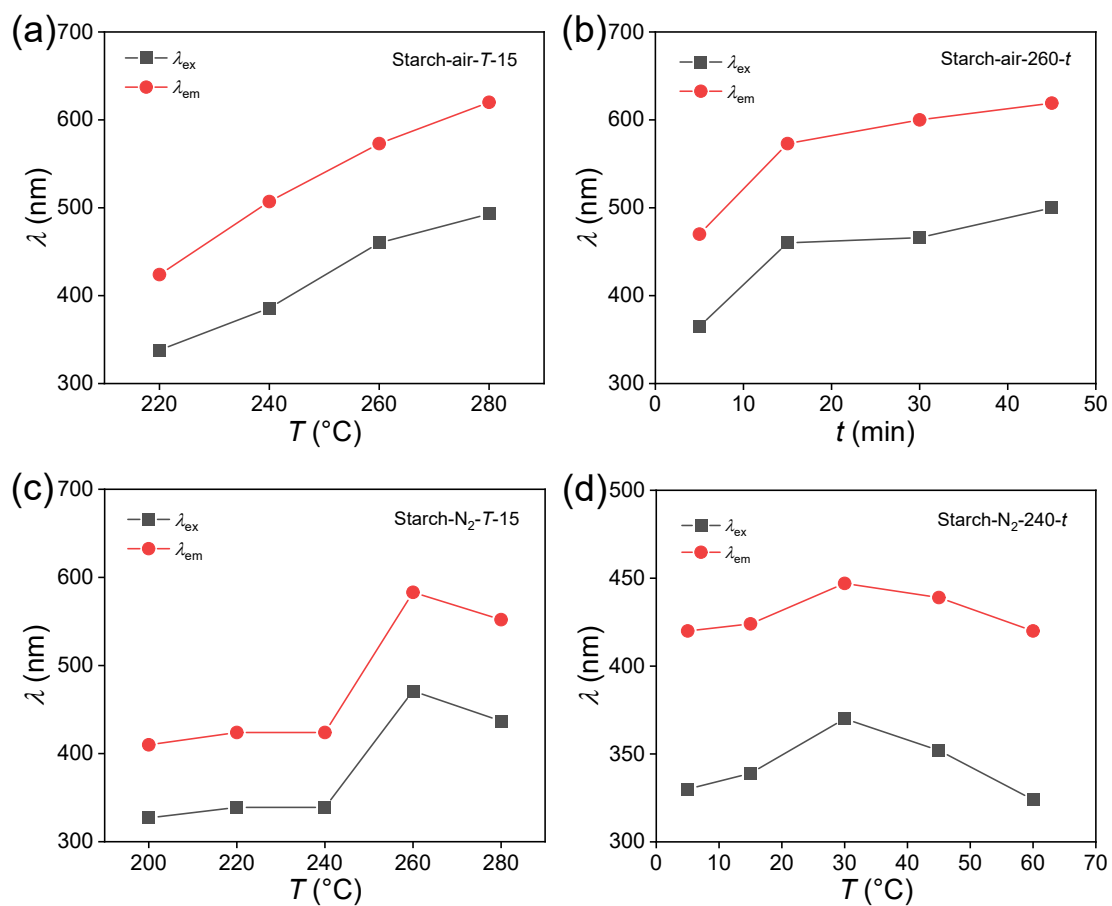




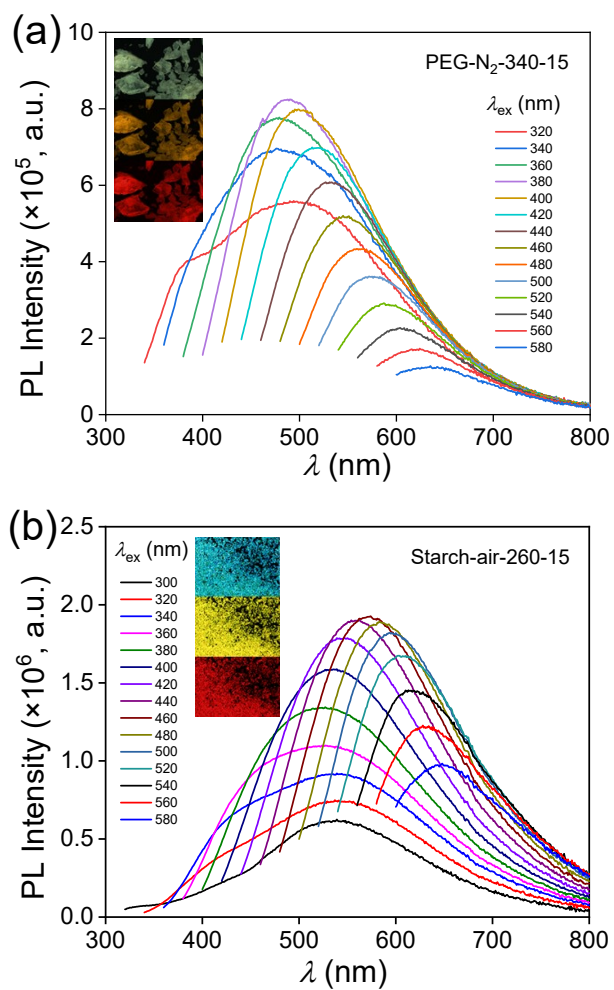
**Fig. S5** The  $\hat{\lambda}_{ex}^{max}$  and  $\hat{\lambda}_{em}^{max}$  of PEG-air-*T*-15 (a), PEG-air-260-*t* (b), PEG-N<sub>2</sub>-*T*-15 (c), and PEG-N<sub>2</sub>-340-*t* (d) solids.



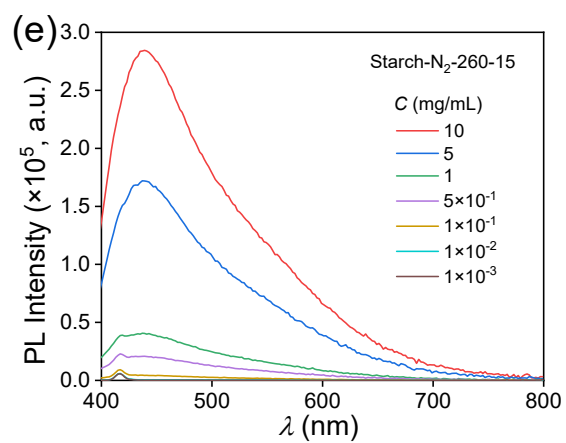
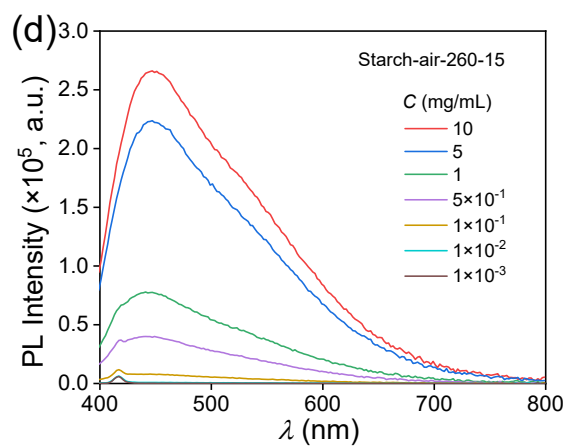
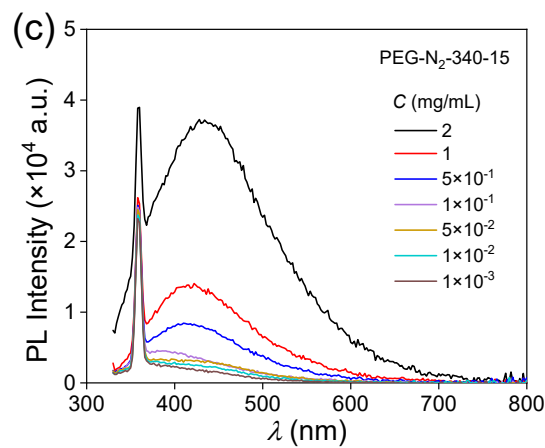
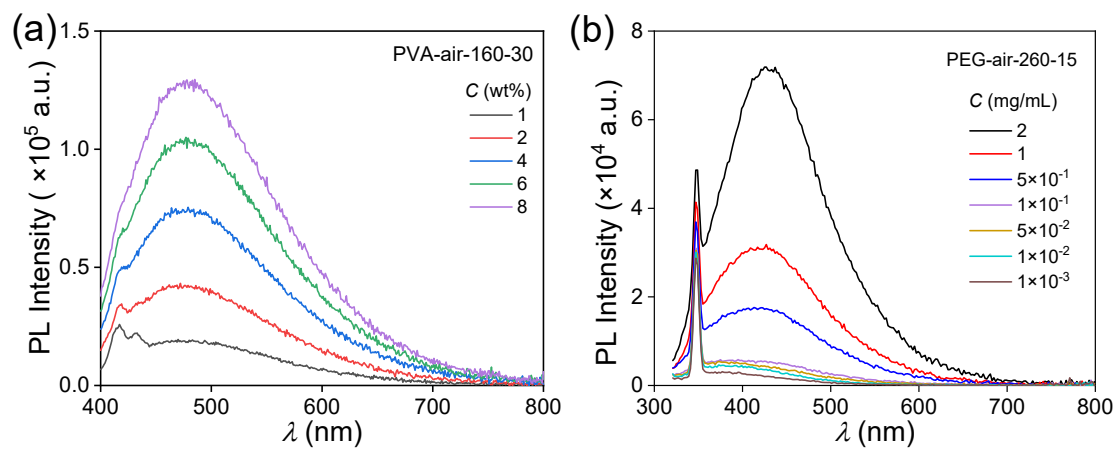
**Fig. S6** Fluorescence spectra of original starch (a) and its gas-thermal product solids obtained under different conditions (b-e).



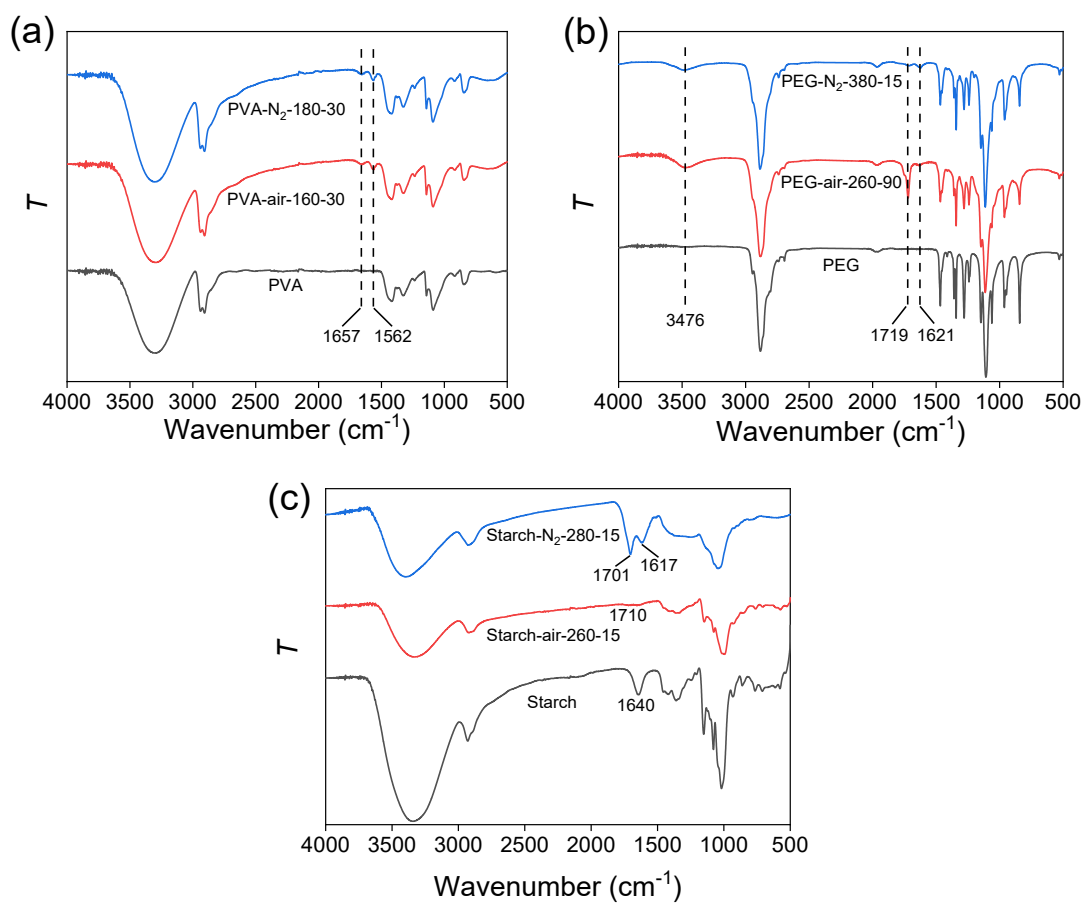
**Fig. S7** The  $\lambda_{ex}^{max}$  and  $\lambda_{em}^{max}$  of starch-air- $T$ -15 (a), starch-air-260- $t$  (b), starch- $\text{N}_2$ - $T$ -15 (c), and starch- $\text{N}_2$ -240- $t$  (d) solids.



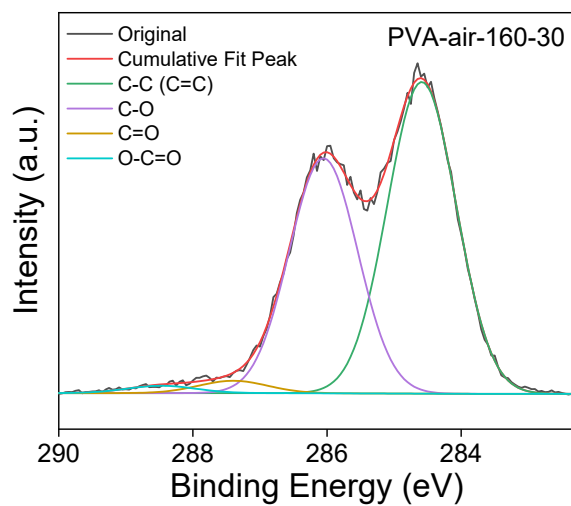
**Fig. S8** Fluorescence emission spectra of PEG-N<sub>2</sub>-340-15 (a) and starch-air-260-15 (b) solids under different excitation wavelengths. The insets are microscopic photos of the corresponding product under the irradiation of UV, blue and green lights (from top to bottom).



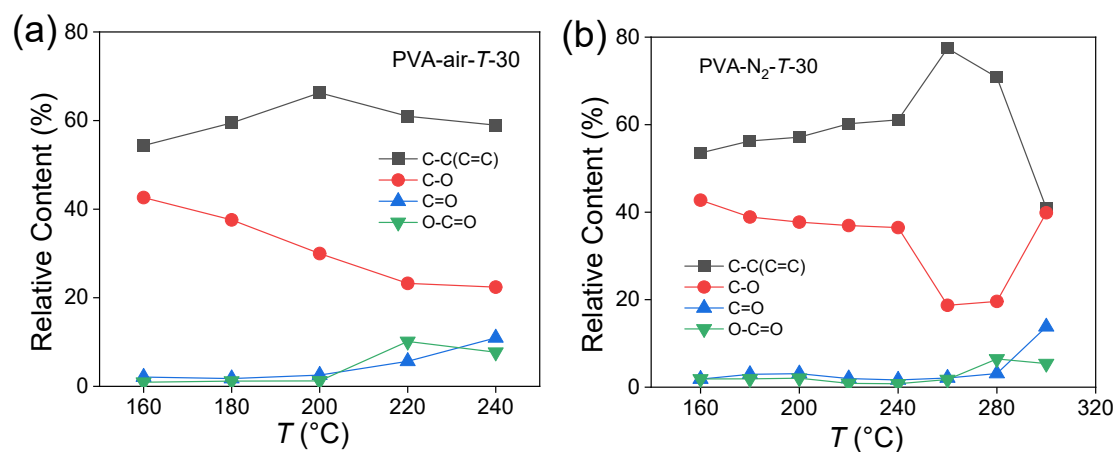
**Fig. S9** Fluorescence emission spectra of aqueous solutions of some gas-thermal products with different concentrations.



**Fig. S10** FTIR spectra of the original and gas-thermal products of PVA (a), PEG (b), and starch (c).

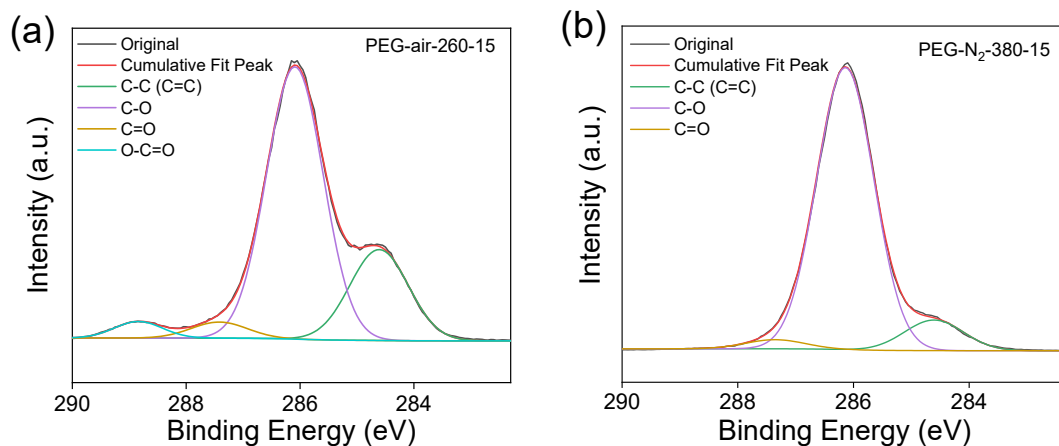


**Fig. S11** Curve-fitted  $C_{1s}$  XPS spectra of PVA-air-160-30.

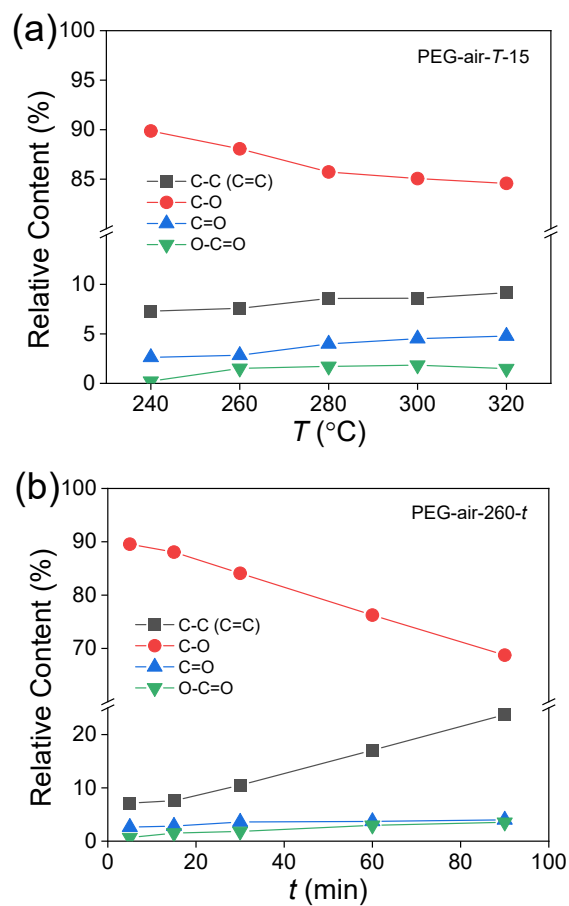


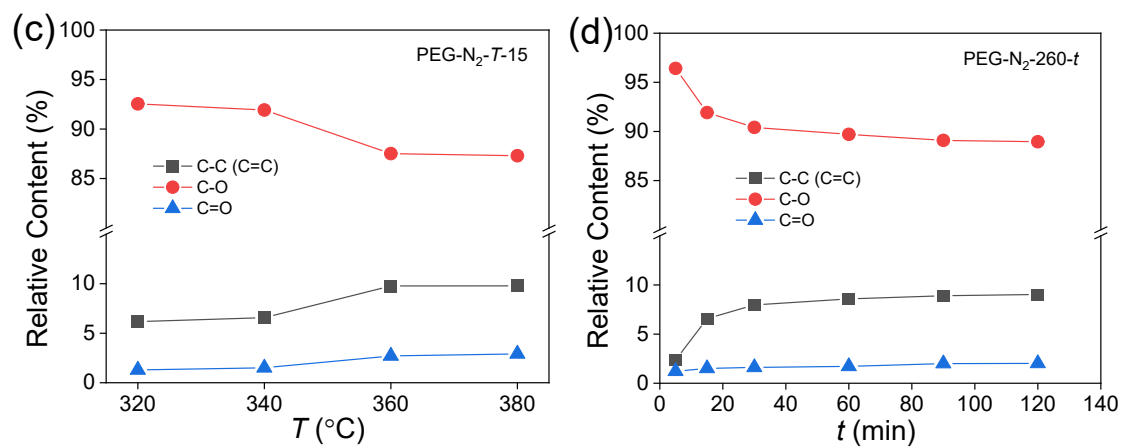
**Fig. S12** Relative contents of different kinds of C in PVA-air- $T$ -30 (a) and PVA- $N_2$ - $T$ -30 (b).



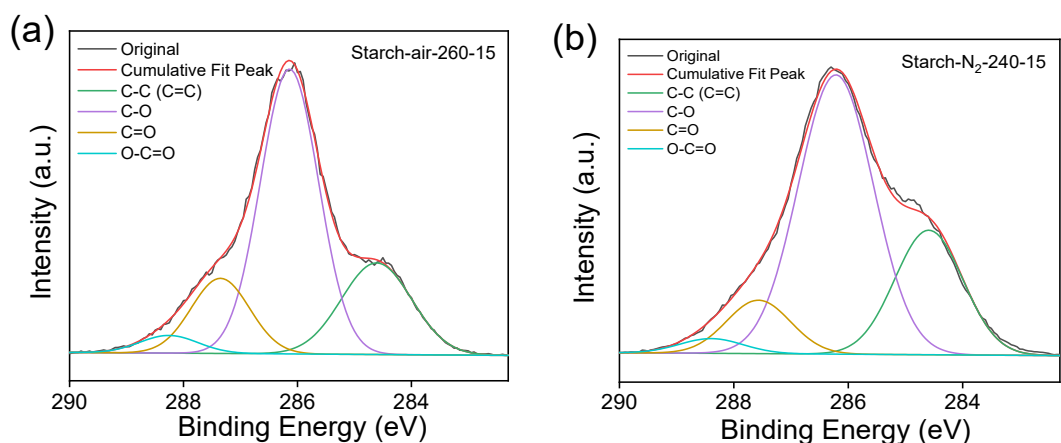


**Fig. S13** Curve-fitted C<sub>1s</sub> XPS spectra of PEG-air-260-15 (a) and PEG-N<sub>2</sub>-380-15 (b).

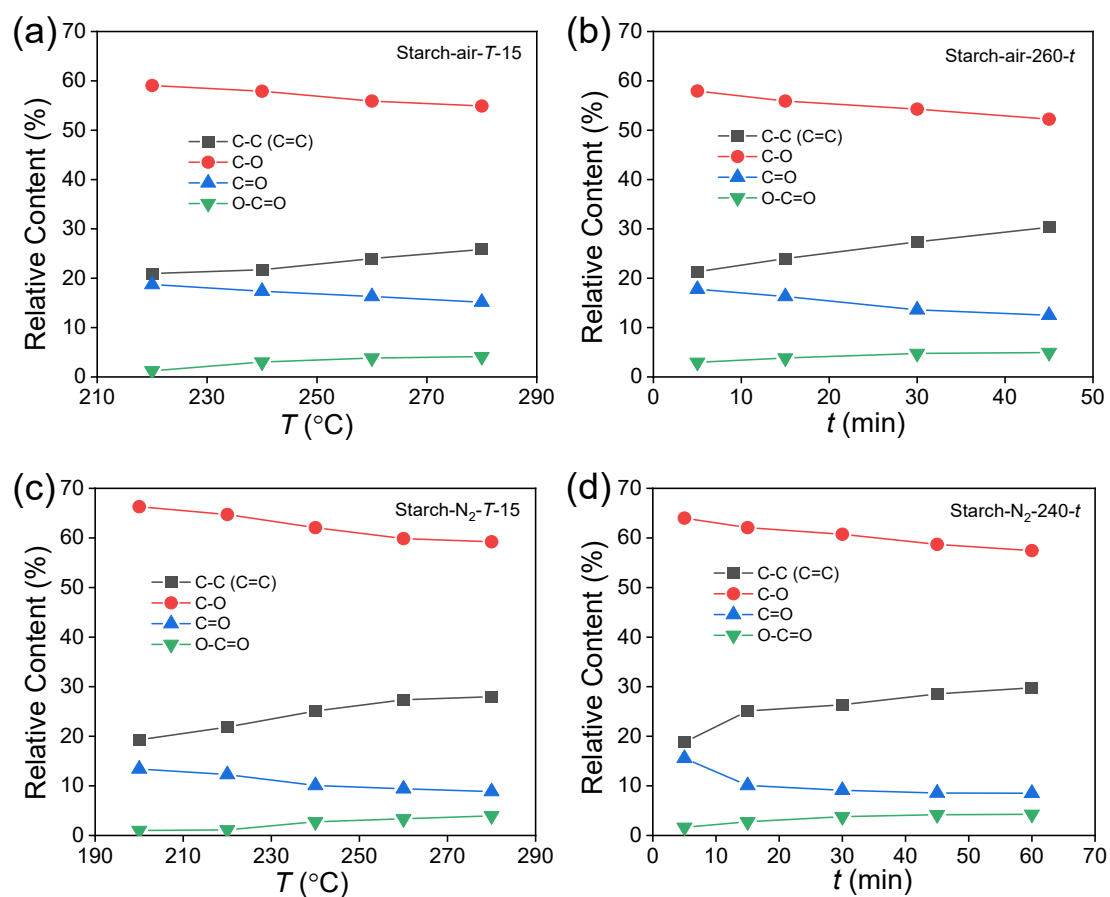




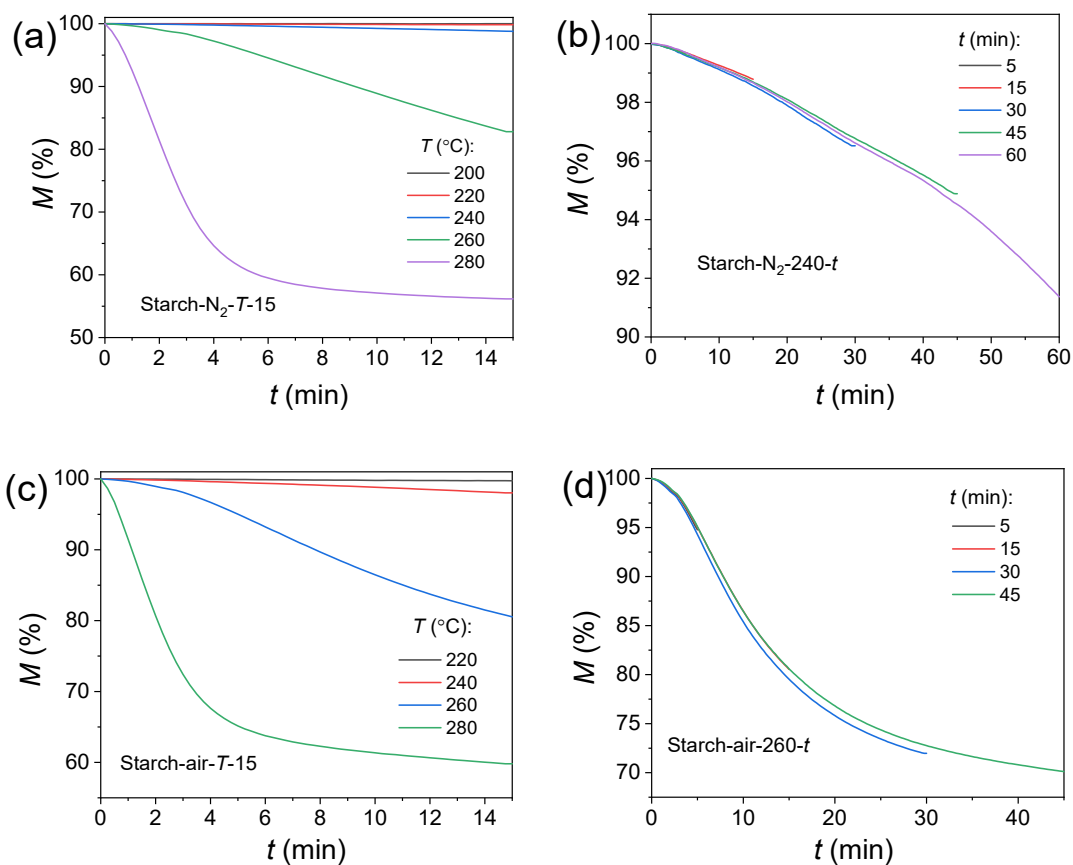
**Fig. S14** Relative contents of different kinds of C in PEG gas-thermal products.



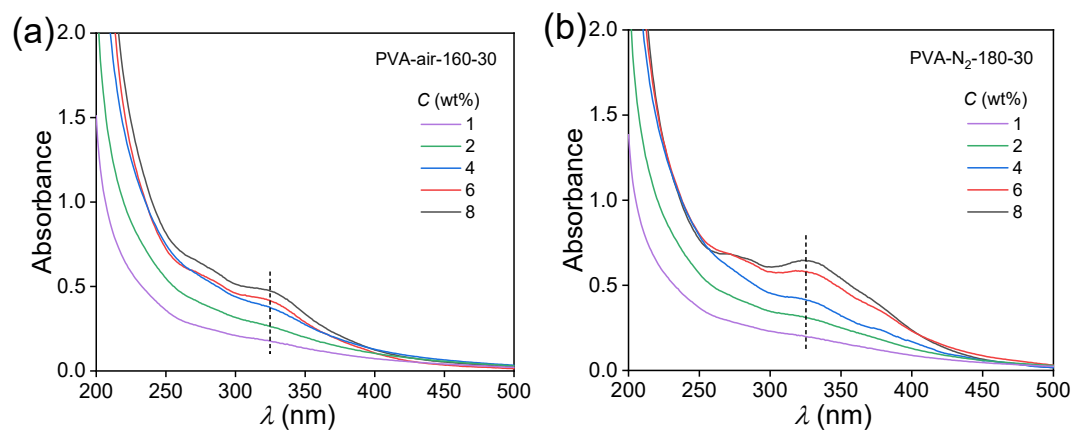
**Fig. S15** Curve-fitted C<sub>1s</sub> XPS spectra of starch-air-260-15 (a) and starch-N<sub>2</sub>-240-15 (b).



**Fig. S16** Relative contents of different kinds of C in starch gas-thermal products.



**Fig. S17** TGA curves of starch heated in nitrogen (a, b) or air (c, d) at different temperatures for 15 min (a, c) and at 240 °C or 260 °C for different times (b, d).



**Fig. S18** UV-vis absorption spectra of PVA-air-160-30 (a) and PVA-N<sub>2</sub>-180-30 (b) aqueous solutions with different concentrations.