

Enhanced thermal stability of polystyrene by graphitic carbon nitride/spinel ZnCo_2O_4 nanohybrids and the catalytic mechanism investigation

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2. Experimental section

2.1. Raw materials

PS (158 K) was obtained from BASF-YPC Co., Ltd. (China). Cobaltous acetate tetrahydrate ($\text{Co}(\text{OAc})_2 \cdot 4\text{H}_2\text{O}$), Zinc nitrate hexahydrate ($\text{Zn}(\text{NO}_3)_2 \cdot 4\text{H}_2\text{O}$), urea, and anhydrous ethanol were purchased from Sinopharm Chemical Reagent Co., Ltd. (Shanghai, China). Ammonium hydroxide ($\text{NH}_3 \cdot \text{H}_2\text{O}$) was provided by Shanghai Pilot Chemical Co., Ltd. (Shanghai, China). All reagents were used as received without further purification.

2.2. Preparation of $\text{ZnCo}_2\text{O}_4/\text{g-C}_3\text{N}_4$ nanohybrids

The bulk $\text{g-C}_3\text{N}_4$ was synthesized according to our previous work.¹ The C- ZnCo_2O_4 nanohybrids were prepared by conventional solvothermal method. In a typical procedure, 0.23 g of as-synthesized $\text{g-C}_3\text{N}_4$ was dispersed in 60 mL of anhydrous ethanol under ultrasonication. After sonication for 2 h, 12 mL of 0.1 M $\text{Co}(\text{OAc})_2$ and 6.25 mL of 0.1 M $\text{Zn}(\text{NO}_3)_2$ were introduced into the suspension above, followed by the addition of 1.75 mL of $\text{NH}_3 \cdot \text{H}_2\text{O}$ at room temperature with further stirring for 10 h. Then the reaction mixture was transferred to a 100 mL of Teflon-lined autoclave and heated at 150 °C for 10 h. The resulting product was collected by centrifugation and washed with ethanol, and dried at 80 °C. The resulting nanohybrid was collected and labeled as C- ZnCo_2O_4 . For comparison, free ZnCo_2O_4 nanoparticles were synthesized by the same strategy as C- ZnCo_2O_4 except for adding $\text{g-C}_3\text{N}_4$.

2.3. Preparation of PS composites

PS composites were prepared by mixing PS matrix with C- ZnCo_2O_4 nanohybrids (2 wt% in this work) in a Brabender-like apparatus (LH-60, afforded by Shanghai Kechuang Plastic Machinery Co., Ltd., China) with a roller speed of 60 r/min at 180 °C for 15 min. The resulting composites were hot-pressed into sheets for characterization. Both of PS/ $\text{g-C}_3\text{N}_4$ and PS/ ZnCo_2O_4 composites were prepared in a similar procedure.

2.4. Characterization

Powder X-ray diffraction (XRD) patterns were performed with a Japan Rigaku Dmax

X-ray diffractometer equipped with graphite monochromatized high-intensity Cu-K α radiation ($\lambda= 1.54178 \text{ \AA}$). Transmission electron microscopy (TEM) was provided by JEOL 2010F with an acceleration voltage of 200 kV. XPS spectra were recorded using a Kratos Axis Ultra DLD spectrometer employing a monochromatic Al K α X-ray source ($h\nu= 1486.6 \text{ eV}$). The morphologies of the fracture surface of PS composites were investigated by SEM (AMRAY1000B, Beijing R&D Center of the Chinese Academy of Sciences, China). These samples were fractured in liquid nitrogen and sputter coated with a gold layer before observation. Thermogravimetric analysis (TGA) of the obtained samples was carried out with Q5000 thermal analyzer (TA Co., USA) from 30 °C to 800 °C at a heating rate of 20 °C min⁻¹ in air atmosphere (flow rate of 100 mL min⁻¹).

References

1. Y. Shi, S. Jiang, K. Zhou, B. Wang, B. Wang, Z. Gui, Y. Hu and R. K. K. Yuen, *RSC Adv.*, 2014, **4**, 2609-2613.