

Fabrication of Metal aminotrimethylene phosphonate and its flame retardant performance in epoxy resin

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Introduction

- Epoxy resin (EP), the most commonly used thermosetting polymer materials, has been widely utilized in various fields such as the **chemical industry, casting, aviation and others**¹. However, the **inherent flammability** of EPs has severely limited their wider applications².
- Aminotrimethylenephosphonic acid (ATMP)** is a widely used and inexpensive water treatment agent used in circulating water systems in refineries and thermal power stations to prevent scale formation. The presence of the P-C bond makes it more stable than inorganic phosphoric acid and easier to transport and use.
- Graphitic carbon nitride (g-C₃N₄)**, one of the common **two-dimensional nanomaterials** that have attracted extensive attention in the preparation and application of flame-retardant polymer composites due to its excellent **thermal stability** and unique **physical barrier effect**³.
- Multiple mechanisms act synergistically⁴ in the **condensed phase** and **gas phase**, leading to improved flame retardancy.

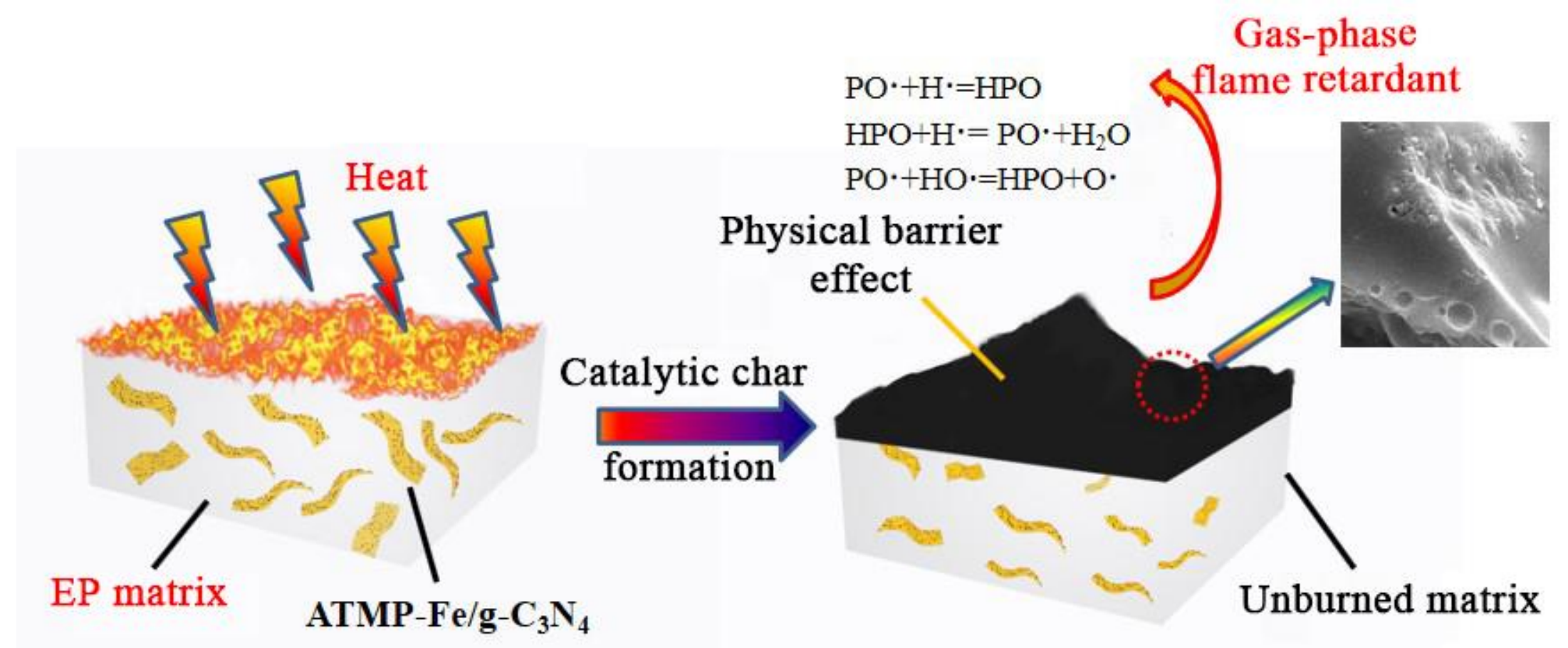


Figure.1 Flame retardant mechanism of ATMP-Fe/g-C₃N₄

Materials and methods

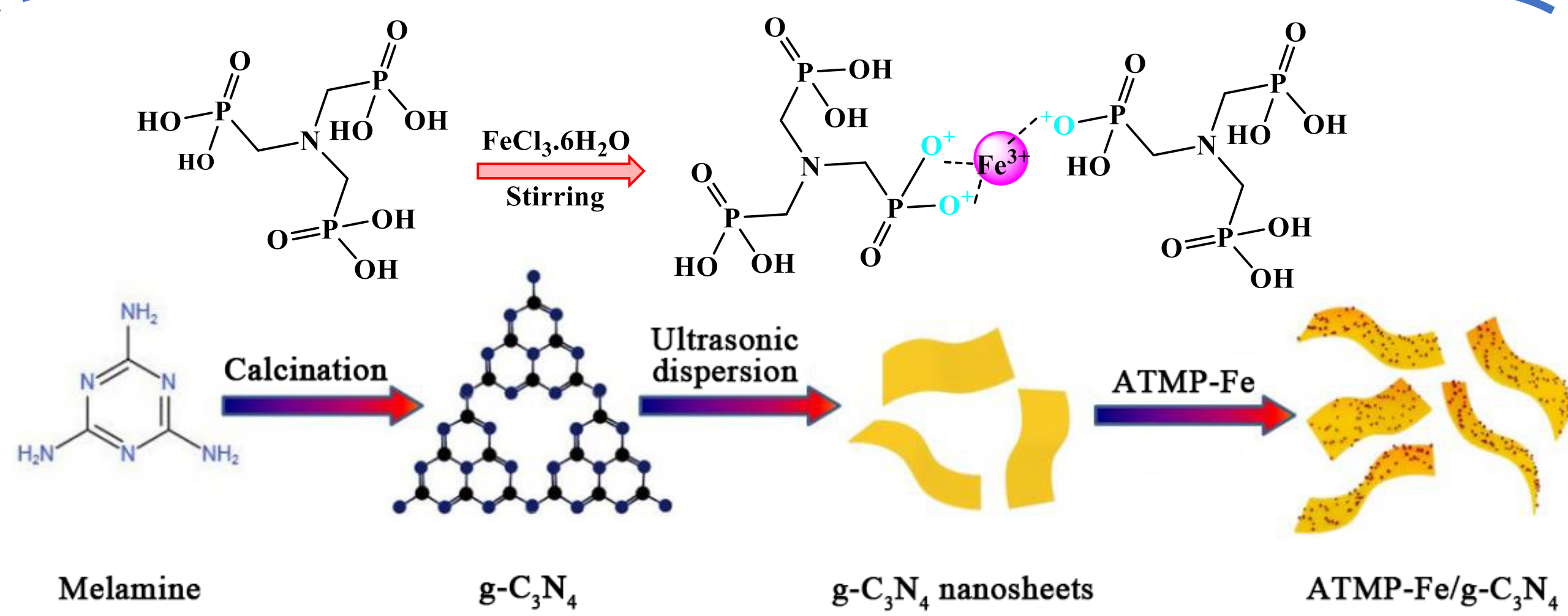


Figure.2 Synthesis route of ATMP-Fe/g-C₃N₄

- Structure:** Fourier transform infrared (FTIR) spectroscopy, X-ray diffraction (XRD) pattern and X-ray photoelectron spectroscopy (XPS).
- Morphology:** Scanning electron microscopy (SEM) and Energy-dispersive X-ray spectroscopy (EDS).
- Thermal stability:** Thermo-gravimetric (TG) analysis and Thermogravimetric analysis-infrared spectroscopy (TG-IR).
- Flame retardancy:** Cone calorimeter test (CCT), Raman spectrometry (LRS) and Scanning electron microscopy (SEM).

Results and discussions

1. Structure

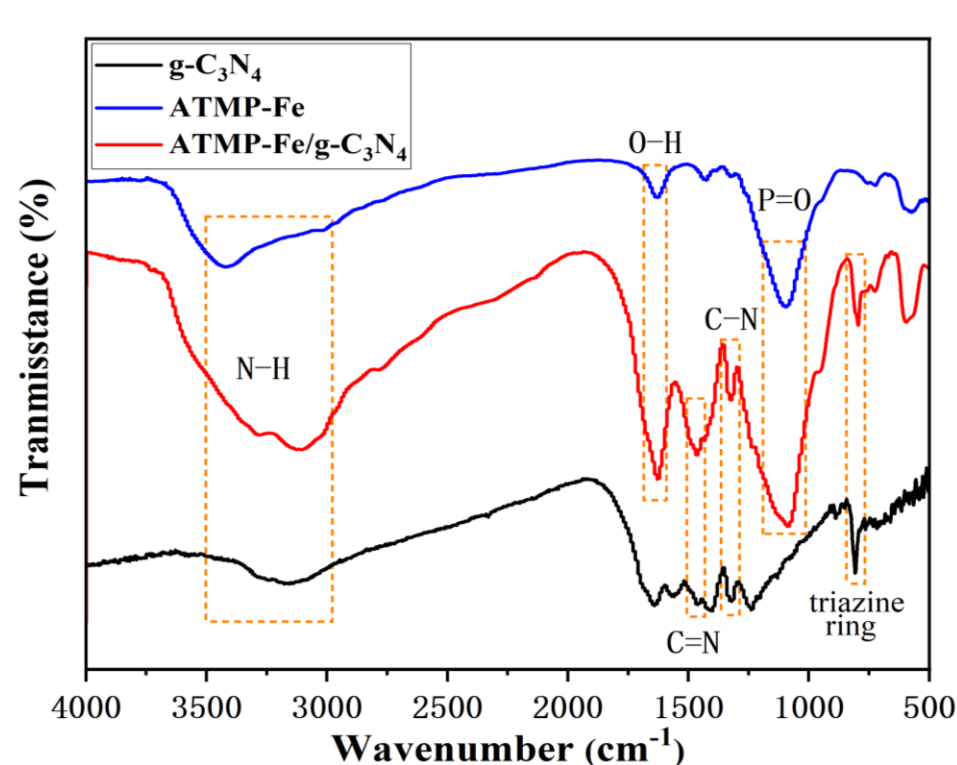


Figure.3 FTIR spectra of ATMP-Fe, g-C₃N₄ and ATMP-Fe/g-C₃N₄

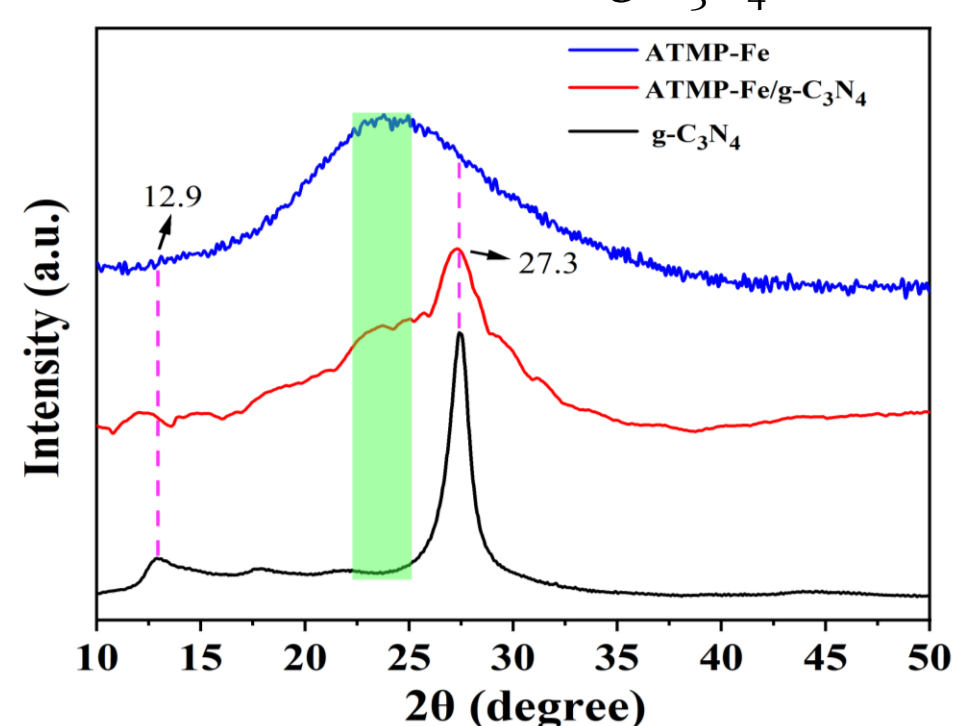


Figure.4 XRD patterns of ATMP-Fe, g-C₃N₄ and ATMP-Fe/g-C₃N₄

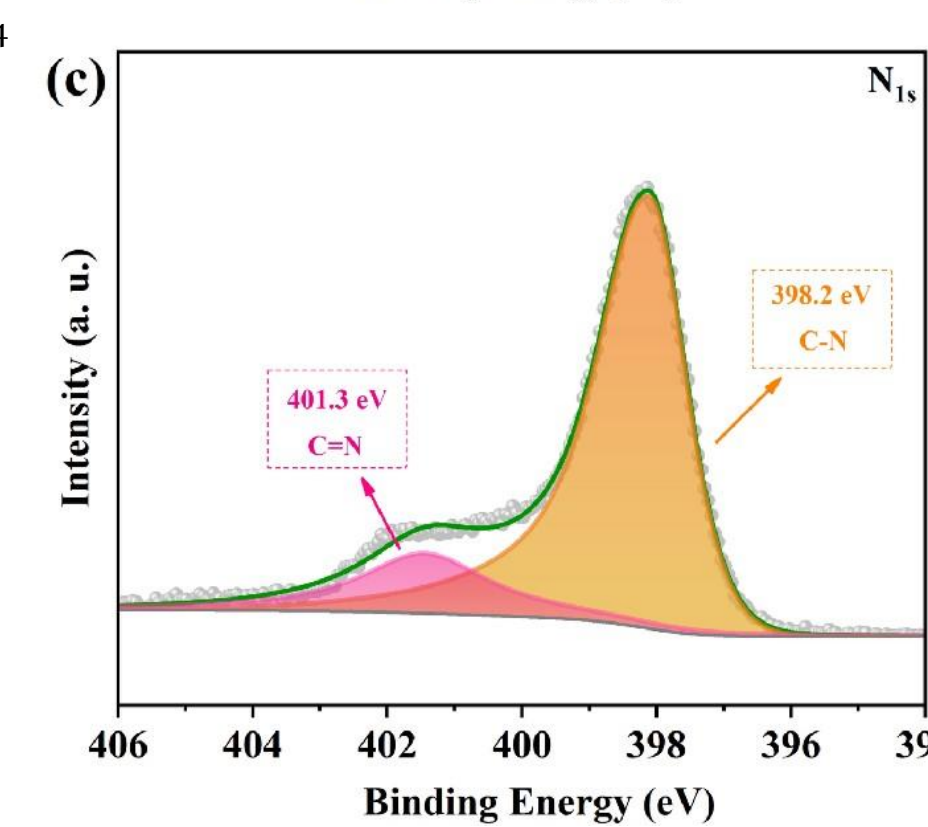
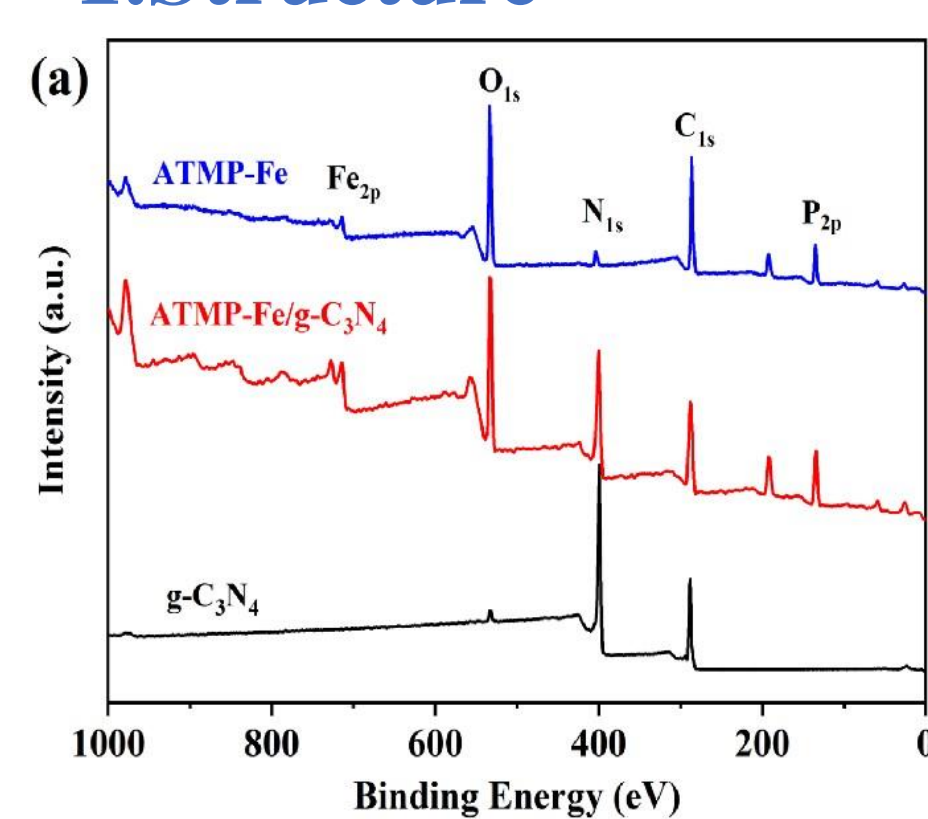


Figure.5 XPS spectra of (a) whole spectra of ATMP-Fe, g-C₃N₄ and ATMP-Fe/g-C₃N₄, (b) C_{1s}, (c) N_{1s} and (d) P_{2p} spectrum of ATMP-Fe/g-C₃N₄

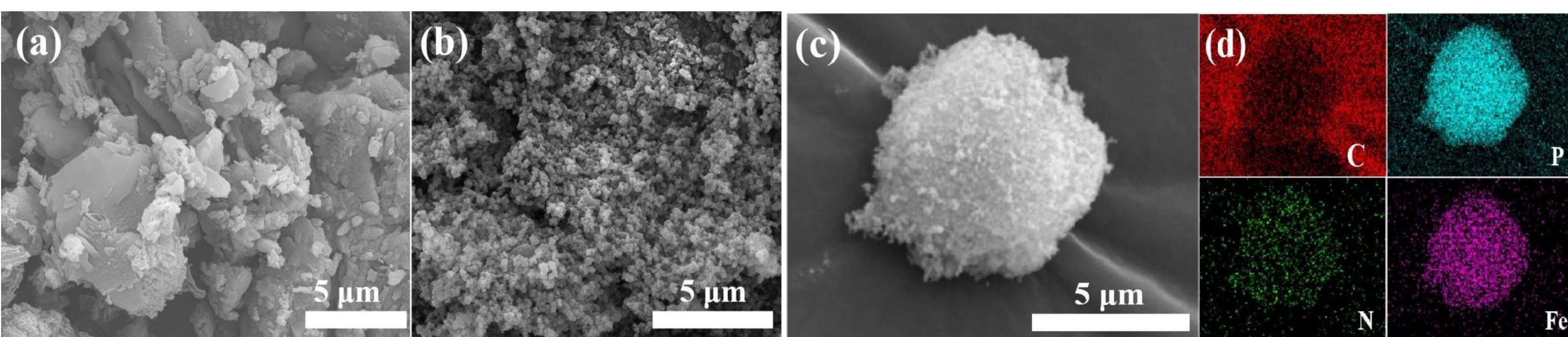


Figure.6 SEM images of g-C₃N₄ (a), ATMP-Fe (b) and ATMP-Fe/g-C₃N₄ (c); EDS mapping of ATMP-Fe/g-C₃N₄ (d)

Acknowledgments

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2. Performance

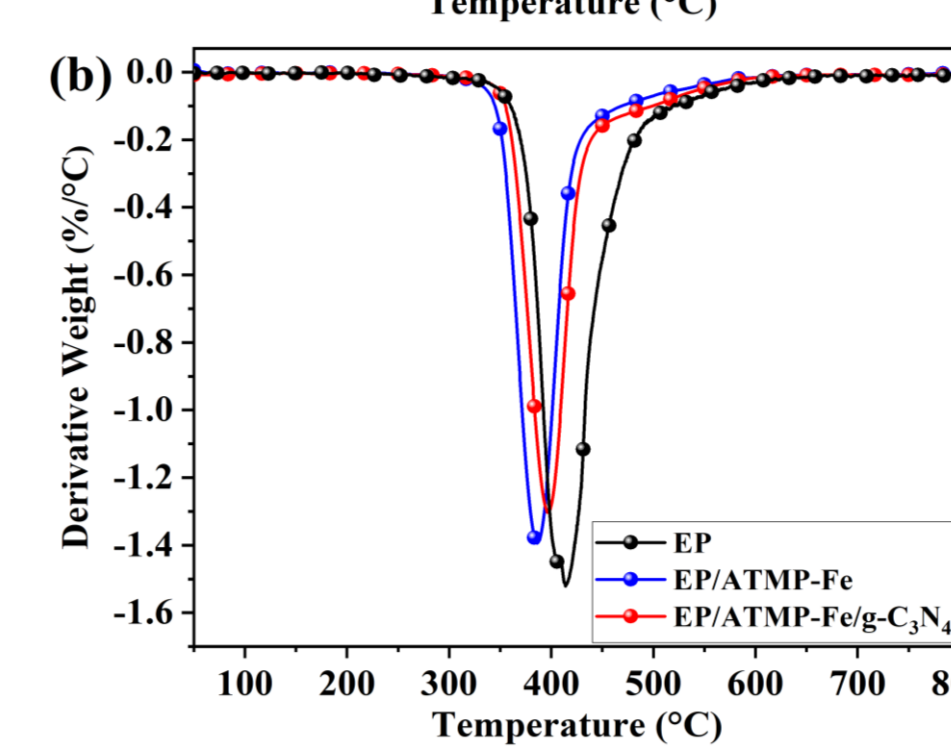
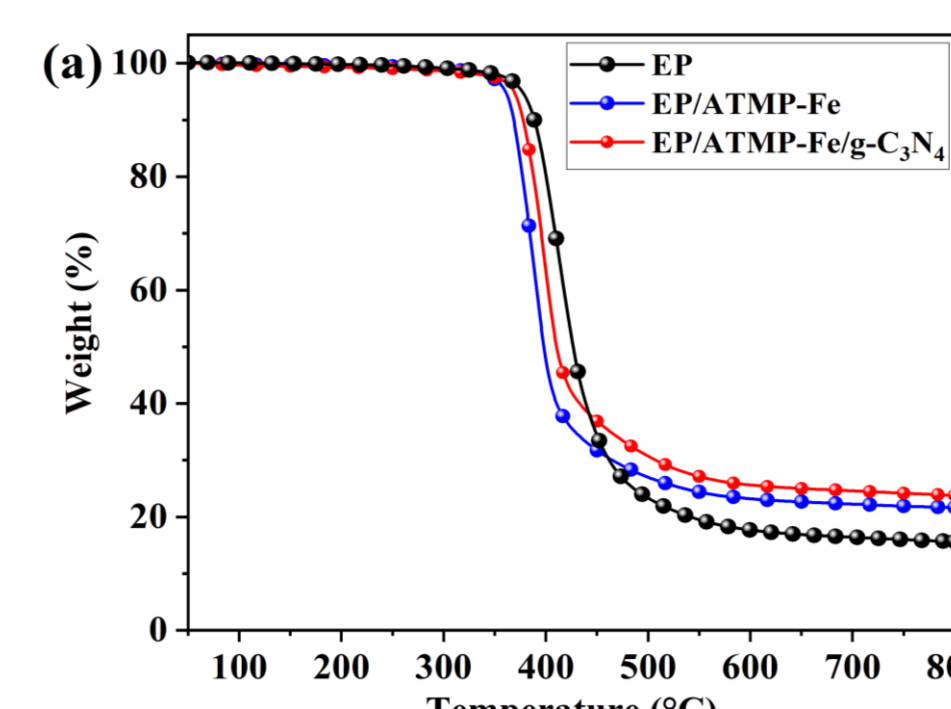


Figure.7 TG (a) and DTG (b) curves of EP, ATMP-Fe, and ATMP-Fe/g-C₃N₄ under N₂ atmosphere

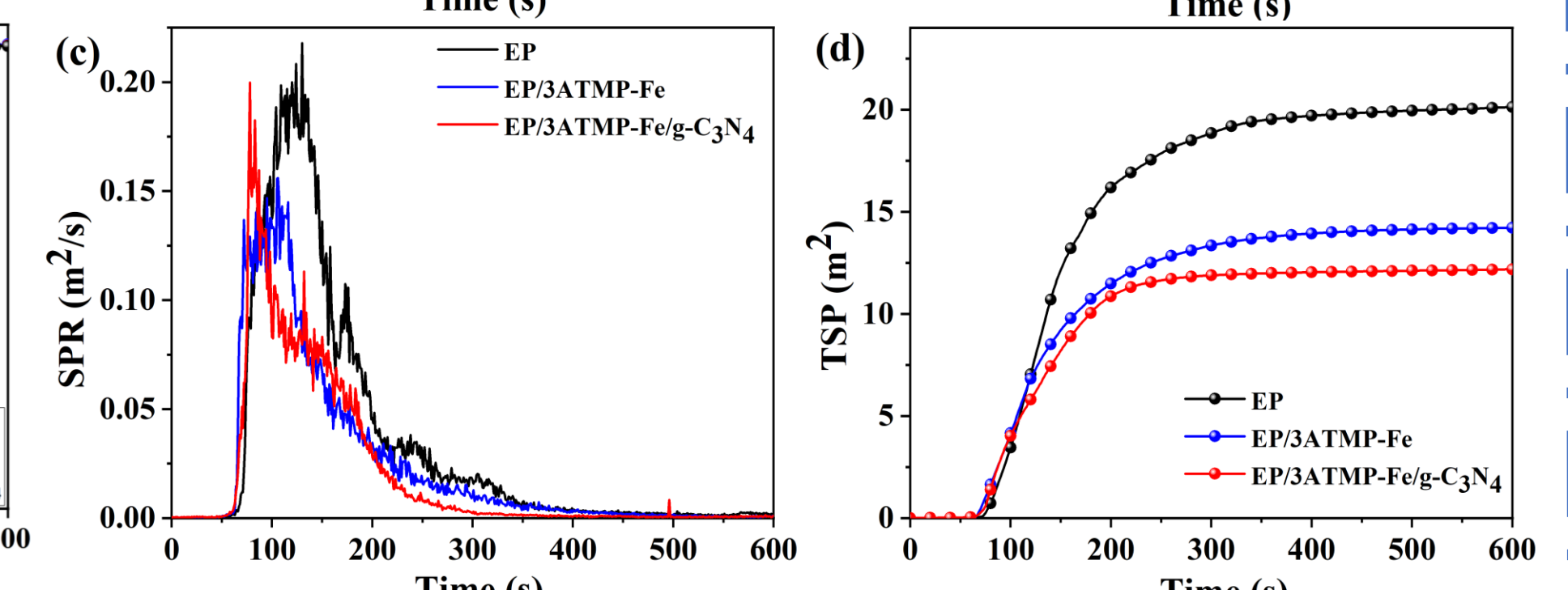
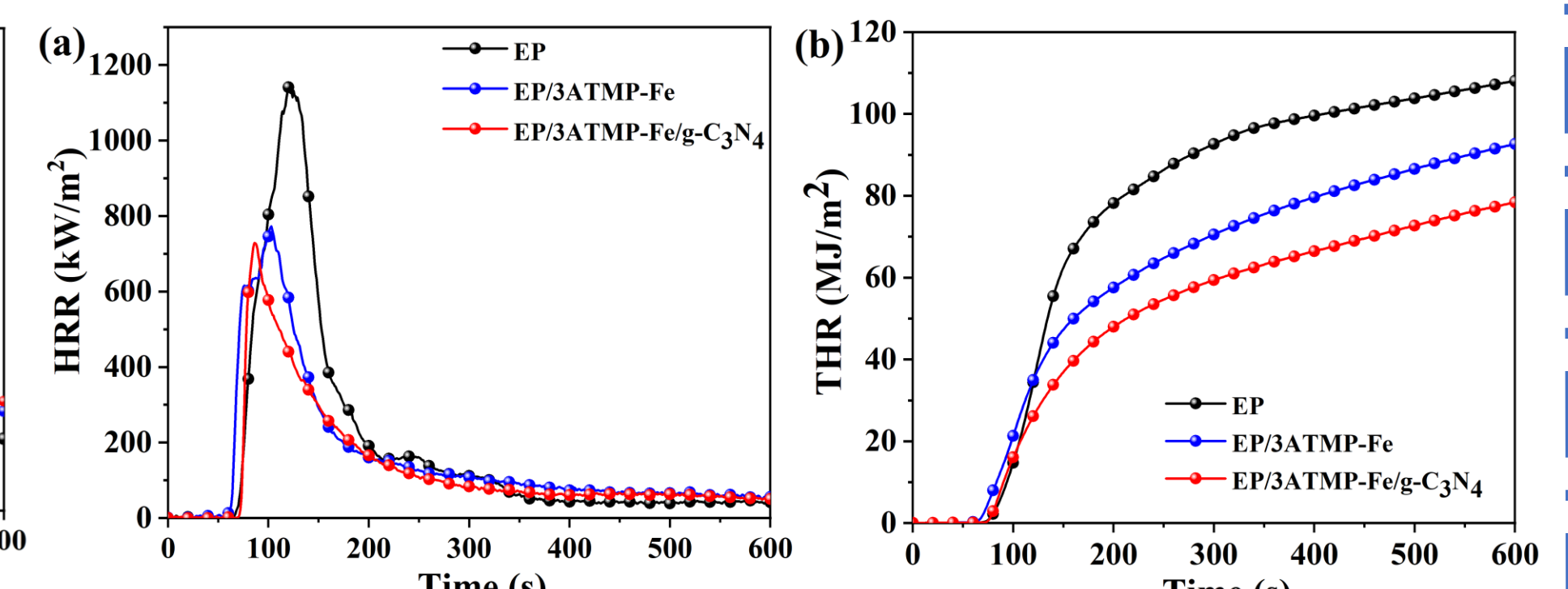


Figure.8 HRR (a), THR (b), SPR (c) and TSP (d) curves of EP and its composites

3. Mechanism

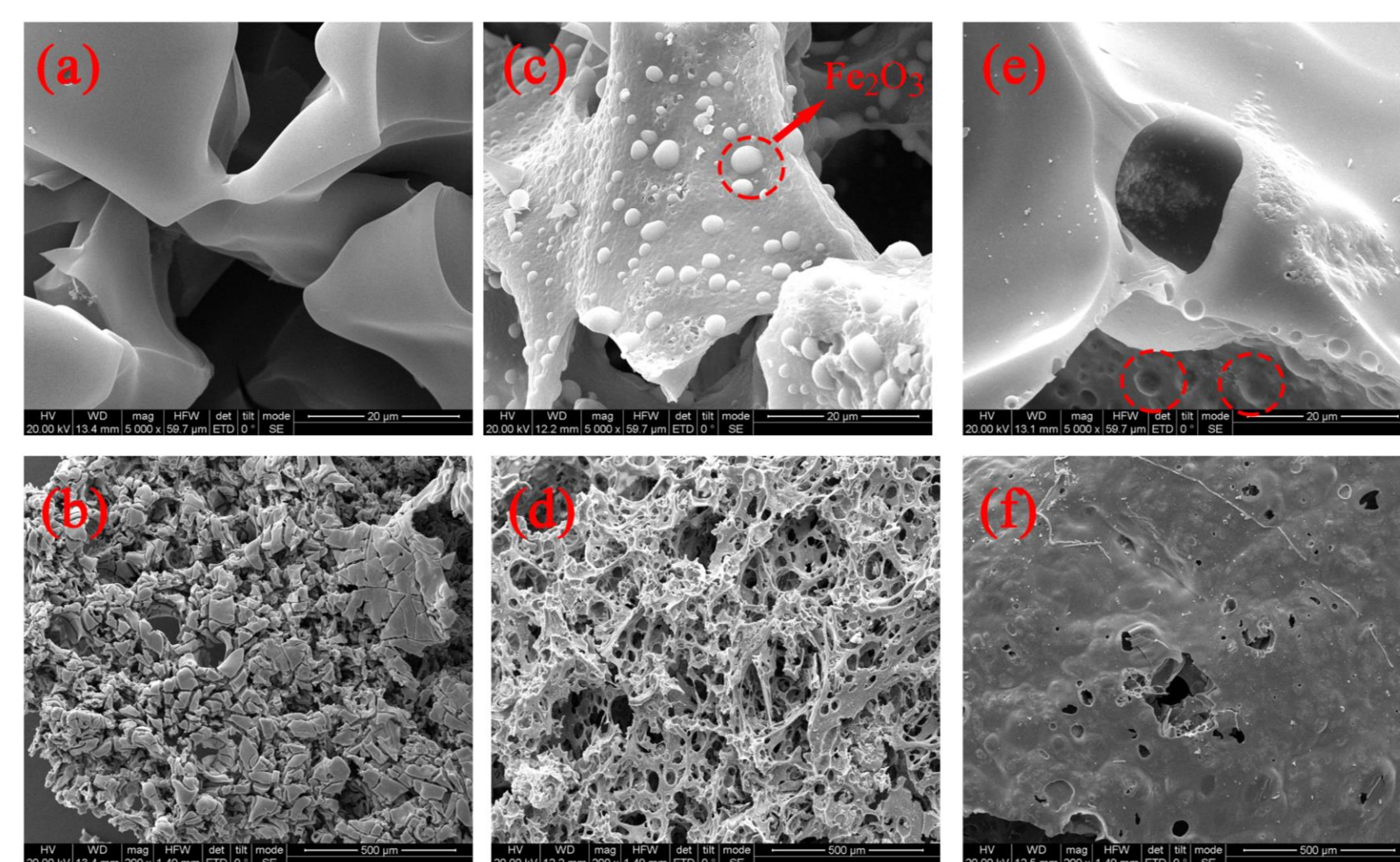


Figure.9 SEM images of char residues of EP (a, b), EP/ATMP-Fe (c, d) and EP/ATMP-Fe/g-C₃N₄ (e, f)

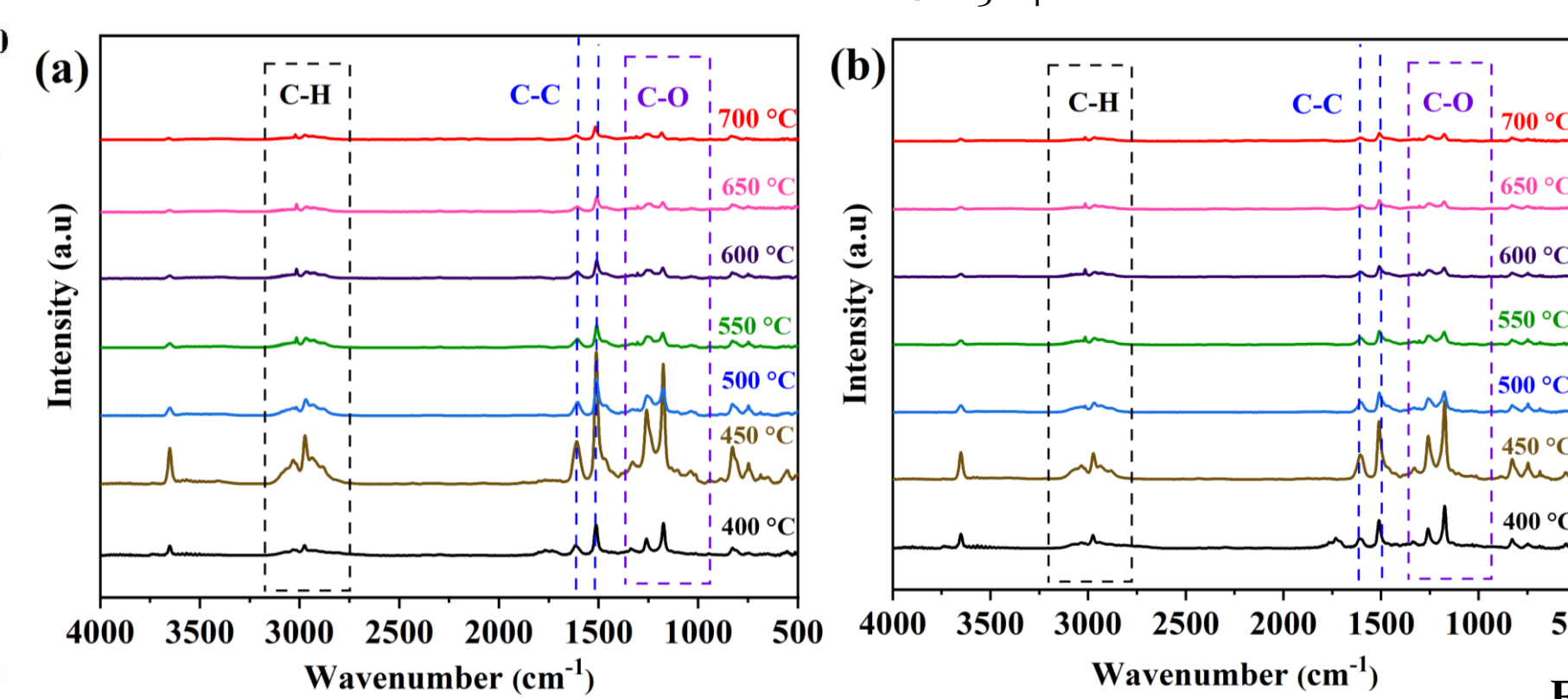


Figure.10 TG-IR curves of EP (a) and EP/ATMP-Fe/g-C₃N₄ (b)

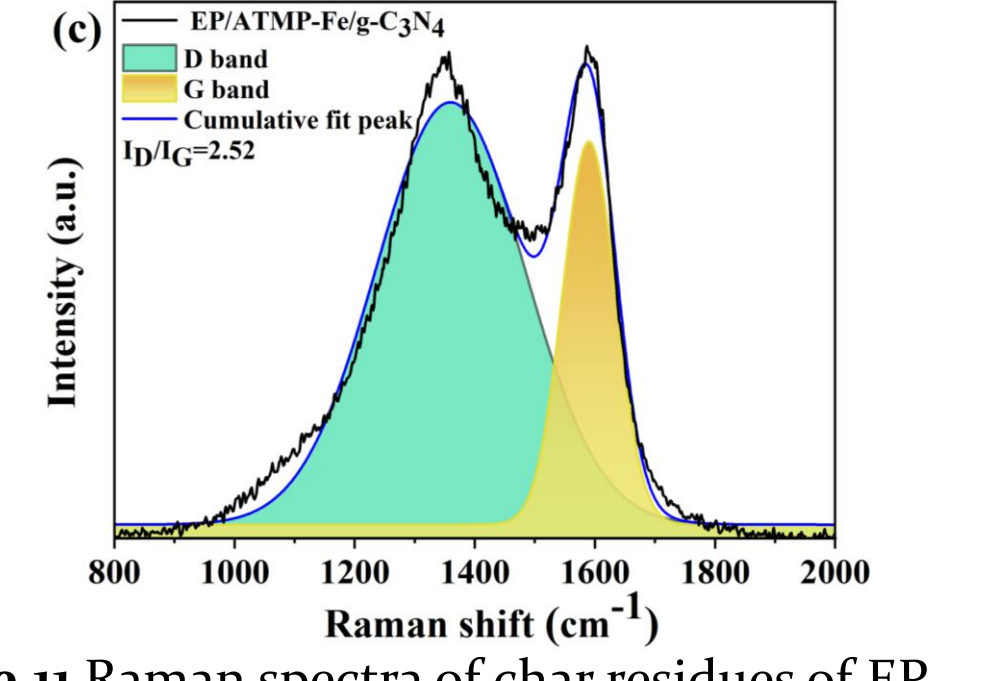
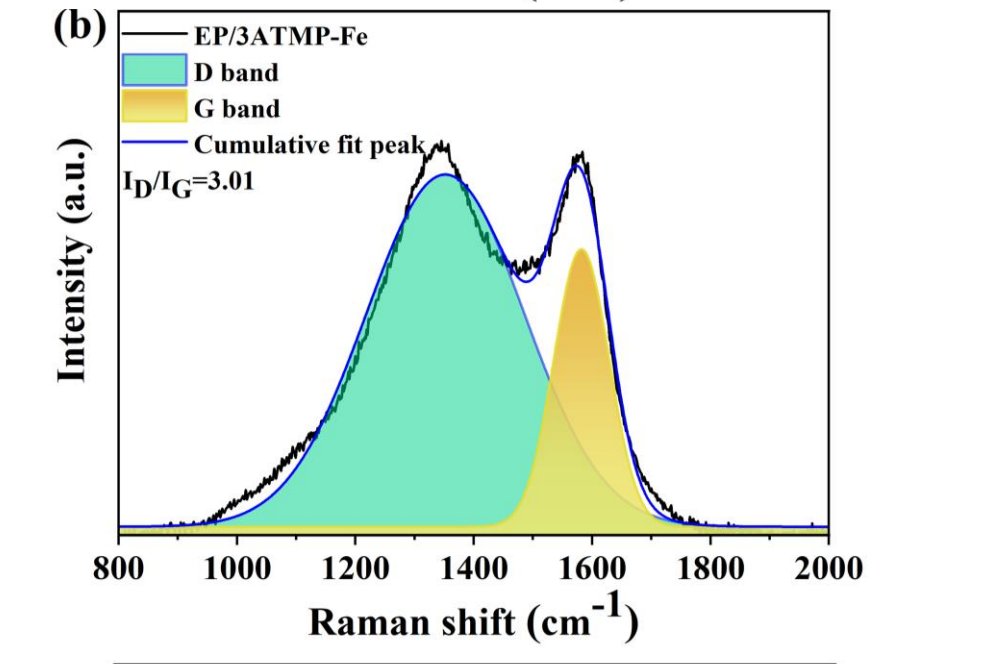
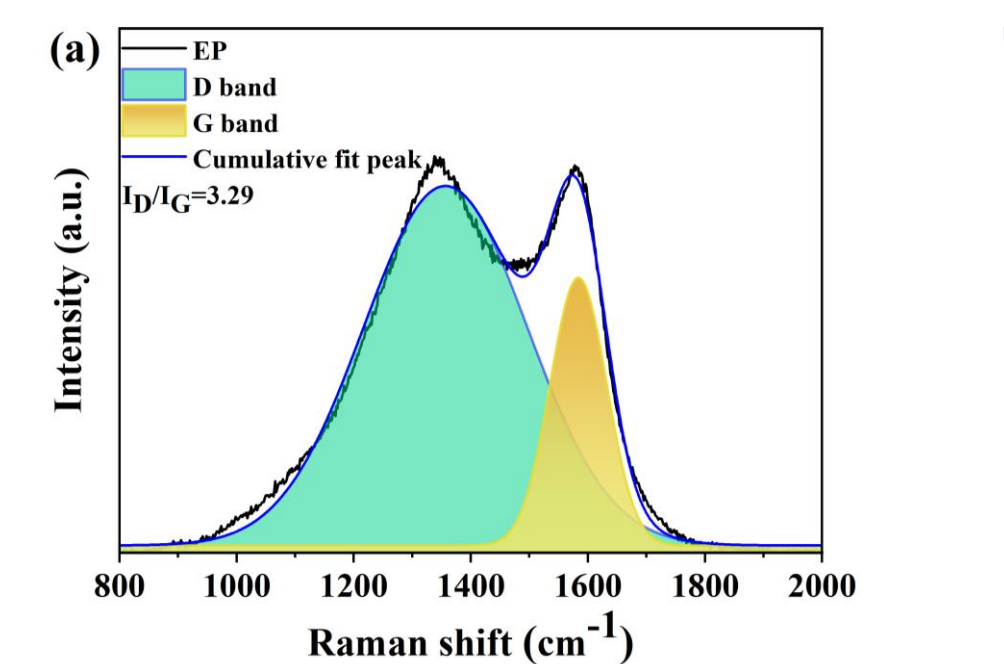


Figure.11 Raman spectra of char residues of EP (a), EP/ATMP-Fe (b) and EP/ATMP-Fe/g-C₃N₄ (c)

Conclusion

Hybrid material (ATMP-Fe/g-C₃N₄) was prepared by precipitation method and successfully characterized by FTIR, XRD and XPS, together with Morphology by SEM and EDS. Then it was added into EP composites. The test results indicated that the addition of 3 wt% ATMP-Fe/g-C₃N₄ can effectively improve the flame retardant efficiency and thermal stability of EP composites. The pHRR, THR and TSP decreased by 36.3%, 27.5% and 39.3%, respectively. The char residues of EP/ATMP-Fe/g-C₃N₄ at 800 °C increased from 15.6% to 23.9%. SEM and LRS results indicated that char residues of EP/ATMP-Fe/g-C₃N₄ were most condensed among all the samples and phosphorus as well as iron participated in the char-forming reaction of EP.

References

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