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Preparation of MES@Fe₃O₄@SiO₂-PPy magnetic microspheres for highly

efficient removal of Cr(VI)

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1. Text S1 experimental details

1.1 Preparation of monodispersed emulsion sphere

The monodispersed emulsion sphere (MES) microspheres were synthesized according to the previous study with several modifications.¹ Styrene (St), Methyl methacrylate (MMA), and Acrylic acid (AA) were stirred evenly in ultra-pure water. Then NH₄·HCO₃ and Sodium dodecyl benzene sulfonate (SDBS) were added while stirring with magnetic force at 70 °C for 30 min. Finally, Ammonium persulfate (APS) was added and the reaction continued for 10 h to prepare MES, which was sealed and stored.

1.2 Preparation of MES@Fe₃O₄ composites

MES@Fe₃O₄ (MF) was synthesized by chemical co-precipitation method. MES, FeCl₂·4H₂O, and FeCl₃·6H₂O were dispersed in ultra-pure water and mixed in N₂ atmosphere for 15 min. After that, NH₃·H₂O was added and the reaction continued for 1 h. The product was repeatedly washed with ultra-pure water until the filtrate was neutral, and then recycled with magnets. Finally, it was dried at 60 °C until constant weight.

1.3 Preparation of MES@Fe₃O₄@SiO₂ composites

MES@Fe₃O₄@SiO₂ (MFS) was synthesized by the Stöber method. Firstly, MES@Fe₃O₄ was dispersed in dilute hydrochloric acid solution for ultrasonic activation for 15 min. Then the activated MES@Fe₃O₄ was added to the mixed solution of absolute ethanol, ultra-pure water, and NH₃·H₂O, and mechanically stirred for 30 minutes. TEOS was slowly added to the above dispersion under stirring. Finally, the sample was recovered by magnet and washed with anhydrous ethanol and ultra-pure water three times each, and dried at 60 °C for 10 h to obtain MFS.

2. Table S1

Sample	MES@Fe ₃ O ₄ @SiO ₂ (g)	Py(g)	FeCl₃ · 6H₂O(g)
MFSP-I	1.0	0.10	0.926
MFSP-II	1.0	0.20	1.852
MFSP-III	1.0	0.30	2.778
MFSP-IV	1.0	0.40	3.704
MFSP-V	1.0	0.50	4.630
MFSP-VI	1.0	0.60	5.556
MFSP-VII	1.0	0.70	6.428

Table S1 Synthesis conditions of MES@Fe₃O₄@SiO₂-PPy

MFSP-VIII	1.0	0.80	7.408
MFSP-IX	1.0	0.90	8.334

3. Figure S1

Fig. S1 showed the DTG plots of PPy, MFS, and MFSP. Obviously, MFS was thermally stable until the temperature of 439 °C. After functionalization of PPy, the decomposition of MFSP nanocomposite occurs at 245 °C. The thermal stability of MFSP was slightly lower than that of MFS in the low temperature region, which might be PPy was easy to volatilize and decompose ².



Fig. S1 DTG curves of PPy, MFS, and MFSP.

Notes and references

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- 2 M. Wysocka-Zolopa, I. Zablocka, E. Gradzka, J. Goclon and K. Winkler, *Microporous Mesoporous Mat.*, 2021, 317, 110954.