

Polyoxometalate-ionic liquid functionalized magnetic nanocomposites for solid phase extraction and HPLC determination of sulfonamides in food samples

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1. Synthesize Well-Dawson POM

Dawson-type polyoxometalate (POM) was synthesized by dissolving 6.25 g of sodium tungstate dihydrate ($\text{Na}_2\text{WO}_4 \cdot 2\text{H}_2\text{O}$) in a mixture of 12 mL of water and 5 mL of 85% orthophosphoric acid. The solution was refluxed for approximately 4 hours. The resulting greenish color was removed by adding a few drops of bromine water to the hot solution. After cooling, 2.5 g of ammonium chloride was added, and the mixture was stirred for 30 minutes. The light-yellow salt obtained was filtered, dissolved in water, and reprecipitated with an additional 2.5 g of ammonium chloride. The solution was stirred again for 30 minutes, filtered, and the precipitate was dissolved in warm water (45 °C). Potassium chloride was then added to the filtrate, and the resulting precipitate was collected on filter paper and dissolved in warm water (80 °C). Upon slow cooling to 20 °C, white needle-shaped crystals of $\text{K}_{14}[\text{NaP}_5\text{W}_{30}\text{O}_{110}]\cdot\text{H}_2\text{O}$ appeared after 4–5 hours and were collected by filtration. The remaining solution was reheated to boiling and filtered again if necessary. Further addition of potassium chloride to the filtrate led to the precipitation of crude α -octa deca tungstophosphate, which was collected and air-dried for 2 days.

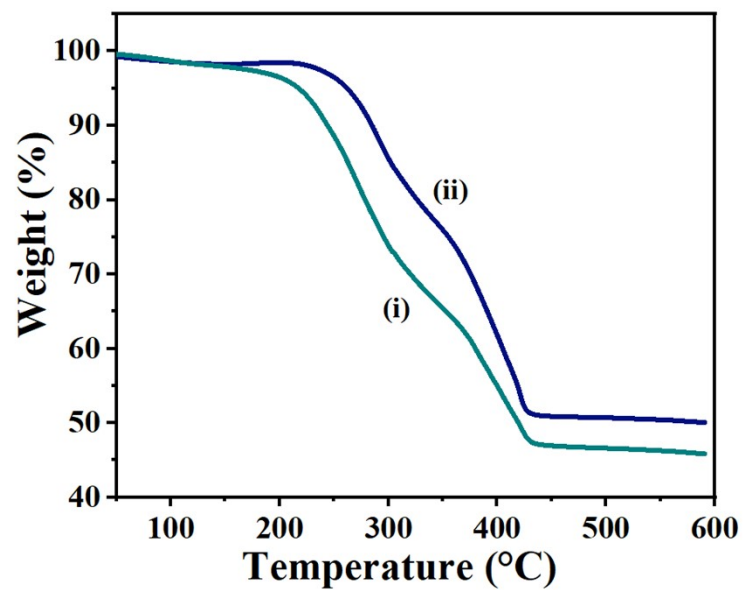


Fig. S1 Thermal gravimetric analysis of MNP@POM-Q⁸IL and MNP@POM-Q¹⁰IL composites.

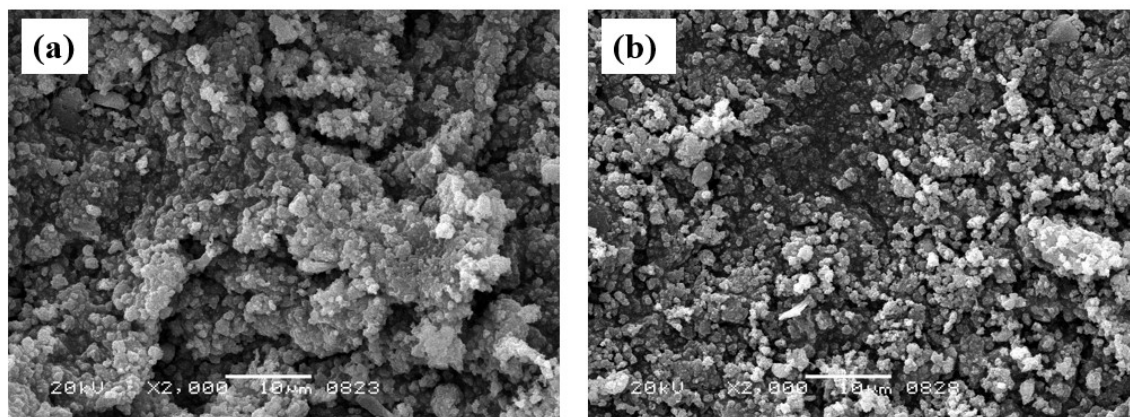


Fig. S2 SEM images showing the rough surface of (a) MNP@POM-Q⁸IL and (b) MNP@POM-Q¹⁰IL.

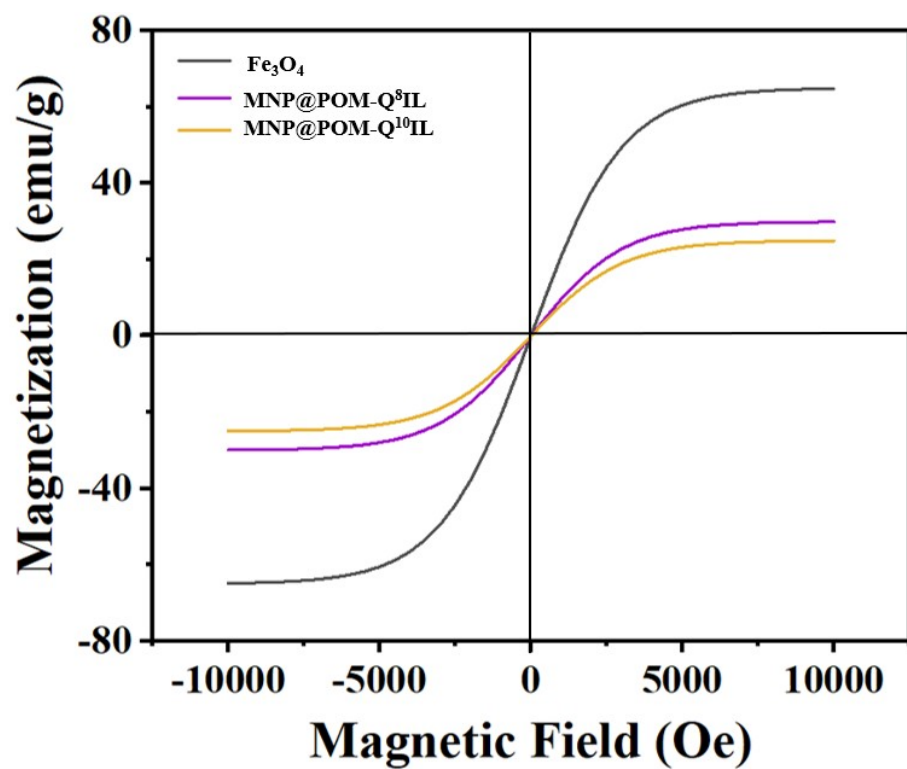


Fig. S3 Vibrating sample magnetometry analysis of Fe_3O_4 , $\text{MNP@POM-Q}^8\text{IL}$, and $\text{MNP@POM-Q}^{10}\text{IL}$ composite