

Super-paramagnetic nano-architecture and its tunable removal efficiency for organic pollutants



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ABSTRACT

In this work, super-paramagnetic nano-architecture was designed to remove the organic pollutants effectively from the waste water. The nano-architectures are composed of super-paramagnetic Fe₃O₄ hollow spheres, conductive polymer and graphene sheets (RGO) to form binary and ternary nano-architectures. They could be magnetically separated and well-distributed into wastewater again for recycle use. Due to the special function of PPy shell, the binary Fe₃O₄@PPy core-shell structure exhibits pH-responsive removal efficiency for organic pollutants. Assembling the Fe₃O₄@PPy core-shell material with RGO sheets could largely improve their surface area, pore volume, and enhance their chemical stability in acid and alkaline solution. Therefore, the Fe₃O₄@PPy@RGO ternary nano-adsorbents could show favorable and robust removal performance for organic pollutants no matter in neutral, acidic and alkaline environment.

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1. Introduction

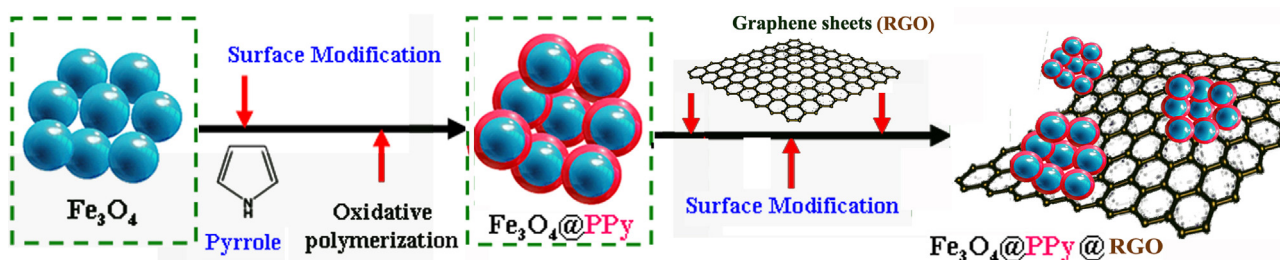
With the rapid development of modern industry, considerable attention has been paid to the environmental problems. Water pollution has become a serious one among those problems [1,2]. The toxic organic dye and heavy metal ions in wastewater not only lead to harmful effect on the human health but on the economic progress. A number of techniques have been employed to remove those hazardous substances from contaminated water, including the reverse osmosis technology, biodegradation, electrochemical treatment, ion exchange method, and adsorption techniques [3–9]. The adsorption technique is recognized to be a simple, economical and environment-friendly way among those techniques [10–14]. Various adsorbents have been reported to remove the pollutants from wastewater, such as clay minerals, zeolites, and transition metal oxides. Furthermore, it has been found that the nano-scaled adsorbents, especially those adsorbents with three-dimensional (3D) nano-structure, show enhanced removal performance than their bulk counterparts [15–20]. The better removal efficiency of nano-scaled adsorbents could be assigned to their large surface area and abundant active sites. However, the practical performance of nano-scaled adsorbents is still limited by two drawbacks [21]. On one hand, the nano-scaled adsorbents tend to agglomerate

because of their high surface energy. That may lead to the decline of absorption efficiency. On the other hand, it is difficult and time-consuming to separate the nano-scaled adsorbents from the solution for recycle use. That may cause potential secondary pollution and add the total cost for wastewater treatment. Therefore, it is desired that the nano-scaled adsorbents could not only have great removal efficiency for the pollutants in water, but could be dispersed well and recycled easily from the solutions.

It remains a challenge to endow the nano-scaled adsorbents with both perfect water-dispersibility and rapid recyclability. Recent researches have focused on the magnetic nano-materials for water treatment due to the convenience of magnetic separation [22,23]. Some magnetic nano-materials exhibit favourable removal efficiency for organic pollutants and heavy metal ions. Their water-dispersibility could be enhanced by modifying them with large amounts of hydrophilic groups. However, the performances of magnetic nano-materials tend to decrease dramatically due to the acid corrosion and surface oxidation. Some efforts have been done to improve the removal stability of magnetic nano-adsorbents. An effective way is to encapsulate those magnetic nano-adsorbents into heterogeneous layers to protect the magnetic core from corrosion in solution [24]. Moreover, in some cases, the core-shell structure could generate fantastic physical and chemical properties due to the interfacial effect between the magnetic core and the functional shell [25–27]. Conductive polymers, such as Polypyrrole (PPy), show potential to be applied as the functional shell due to its good biocompatibility, adjustable electrical and optical properties

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Scheme 1. Synthesis routine of $\text{Fe}_3\text{O}_4\text{@PPy}$ and $\text{Fe}_3\text{O}_4\text{@PPy@RGO}$ nano-architectures.

[28–31]. Some PPy nano-materials could even exhibit pH-sensitive properties [32,33]. The graphene (RGO) sheets are also desirable to protect the magnetic core because of their high mechanical strength and chemical stability. Besides, the graphene sheets are composed of sp^2 -hybridized carbon atoms, which make it able to enhance the removal performance for some organic pollutants via π - π interaction [34–36].

Herein, we report the synthesis and removal properties of three delicate nano-architectures for the removal of an organic pollutant, Methylene blue. As illustrated by Scheme 1, the nano-architectures are composed of these components: (1) Super-paramagnetic Fe_3O_4 hollow spheres with abundant hydrophilic groups; (2) Polypyrrole layers to protect the hollow spheres from acid corrosion and surface oxidation; (3) Graphene sheets to enhance the removal performance of $\text{Fe}_3\text{O}_4\text{@PPy}$ core-shell structure. The super-paramagnetic nano-architectures all show rapid response to magnetic fields. It is convenient to recover such nano-architectures from wastewater by rapid magnetic separation and re-disperse them into water again for recycle use.

2. Experiments

2.1. Preparation of Fe_3O_4 micro-spheres

The mono-dispersed Fe_3O_4 micro-spheres were synthesized by a one-pot hydrothermal method. In a typical synthesis, $\text{FeCl}_3 \cdot 6\text{H}_2\text{O}$ (0.54 g), sodium citrate (1.2 g), and urea (0.2 g) were dissolved in distilled water (40 mL). Then, 0.2 g Polyacrylic acid sodium (PAAS) was added under continuous stirring until it was totally dissolved. The solution was transferred to a 50 mL Teflon-lined autoclave,

which was then sealed and maintained at 200°C for 12 h. The product was separated by magnetic separation, washed with distilled water and absolute ethanol several times, and then dried in a vacuum oven at 60°C overnight.

2.2. Preparation of $\text{Fe}_3\text{O}_4\text{@PPy}$

The $\text{Fe}_3\text{O}_4\text{@PPy}$ were synthesized by oxidative polymerization of pyrrole (Py), as shown in Scheme 1. In a typical synthesis, 0.2 g prepared Fe_3O_4 micro-spheres and 0.05 g poly-vinylpyrrolidone (PVP) (K_{30}) were re-dispersed into 40 mL distilled water via ultrasound. 0.108 g FeCl_3 and 50 mL Py were sequentially added to the solution under vigorous ultrasound vibration. After reacting for 2 h, the product was separated by magnetic separation, washed with distilled water and absolute ethanol several times, and then dried in a vacuum oven at 60°C overnight.

2.3. Preparation of $\text{Fe}_3\text{O}_4\text{@PPy@RGO}$

The $\text{Fe}_3\text{O}_4\text{@PPy@RGO}$ adsorbents were synthesized by hydrothermal method. In a typical synthesis, 0.2 g prepared $\text{Fe}_3\text{O}_4\text{@PPy}$ and 0.05 g PVP (K_{30}) were re-dispersed into 40 mL distilled water via ultrasound. Under vigorous stirring, 1.2 g sodium citrate was dissolved into the above-prepared solution. Then 2 mL graphene oxide (GO) solution (1 mg/mL) was added into above solution subsequently. The solution was transferred to a 50 mL Teflon-lined autoclave, which was then sealed and maintained at 150°C for 6 h. The product was separated by magnetic separation, washed with distilled water and absolute ethanol several times, and then dried in a vacuum oven at 60°C overnight.

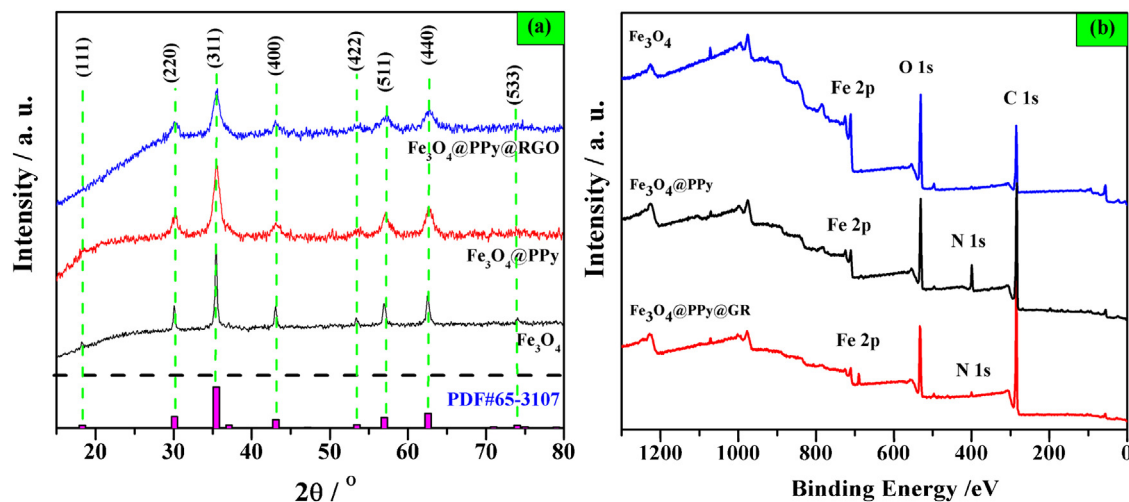


Fig. 1. (a) XRD patterns and (b) XPS survey spectra of Fe_3O_4 , $\text{Fe}_3\text{O}_4\text{@PPy}$ and $\text{Fe}_3\text{O}_4\text{@PPy@RGO}$.

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