



Functional mesoporous carbon nanospheres: Effects of adding magnetic source during different stage

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ABSTRACT

To solve the challenge of preparing magnetic mesoporous carbon nanospheres (MMCNs) with uniform and disperse particles, a series of MMCNs are achieved by a self-assembly procedure in one-pot aqueous solution. Impacts of magnetic source on the morphology and textural property of the as-synthesized MMCNs are systematically investigated by altering their adding time. An extended “two-stage dispersion polymerization” mechanism is employed to explain the formation of MMCNs. Interestingly, adding Fe^{3+} during the particle growing stage can obtain MMCNs-2, exhibiting uniform particle size, large specific surface area, high adsorption capacity and magnetic properties for improving the release of water-insoluble drugs such as Lovastatin (LOV).

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1. Introduction

Functional mesoporous carbon nanospheres with specific properties, such as magnetic performance and providing more specific active sites [1], have been widely concerned, which can satisfy the demands on specific application [2]. Great efforts have been explored to synthesize the functional mesoporous carbon nanomaterials through doping with different metal nanoparticles [3,4]. Recently, Wang fabricated magnetic mesoporous carbon (MMCNs) by the self-assembly of resorcinol-formaldehyde (RF) and F127 under high concentration of HCl, but a multistep procedure such as hydrothermal treatment was involved to avoid the serious aggregation during carbonization [5]. Therefore, the fabrication of MMCNs with thermal stability and spherical morphology still remains a great challenge.

Herein, we present an aqueous self-assembly route in one-pot to fabricate MMCNs under high acid condition, which can enhance the interaction between carbon precursor and template stemming from the $\text{I}^+\text{X}^-\text{S}^+$ mechanism to avoid the macroscopic phase separation. The cooperation of Fe^{3+} with RF can also be explained via $\text{I}^+\text{X}^-\text{S}^+$ mechanism by using negative chloride ions (Cl^-) as a mediator. Adding magnetic source at different stage and their effects on the MMCNs' morphology and textural property are systemically examined.

2. Materials and methods

For a typical preparation of MMCNs-1, $\text{Fe}(\text{NO}_3)_3 \cdot 9\text{H}_2\text{O}$ (1.5 g) and F127 (0.42 g) were first dissolved in mixed solution (40 mL) including HCl (6.7 mL), isopropanol (5 mL) and water (28.3 mL) at 30 °C. Subsequently, resorcinol (0.22 g) and formaldehyde (0.12 mL) were added. After 6 h, the mixed solution was stirred at 50 °C for about 24 h, filtered, washed and dried. The obtained polymer nanoparticles (PNs) were carbonized at 410 °C for 3 h and then at 800 °C for 3 h under N_2 . Finally, the MMCNs-1 was obtained. For comparison, mesoporous carbon nanospheres (MCNs) were fabricated with no $\text{Fe}(\text{NO}_3)_3 \cdot 9\text{H}_2\text{O}$. The MMCNs-2 and -3 represented that the Fe^{3+} was incorporated by redispersing the PNs in the $\text{Fe}(\text{NO}_3)_3 \cdot 9\text{H}_2\text{O}$ aqueous solution (1 mol/L) and the MCNs-410 °C in 10 mL of ethanol solution containing 1.5 g of $\text{Fe}(\text{NO}_3)_3 \cdot 9\text{H}_2\text{O}$, respectively. A series of MMCNs were defined as MMCNs-x (x represented 1, 2, 3).

3. Results and discussion

As seen in Fig. 1 inset, all samples show a type-IV curve with a distinct hysteresis loop at P/P_0 0.4–0.8 except MMCNs-3, confirming the mesoporous structure consistent with the corresponding PSD curves. However, a small hysteresis loop at P/P_0 0.8–1.0 in the nitrogen sorption isotherm and an ambiguous peak in the PSD curve are observed for MMCNs-3 (Fig. 1d inset), which might result from the interstitial space of the nanoparticles [6]. The disappearance of mesopores can be explained that the Fe_3O_4

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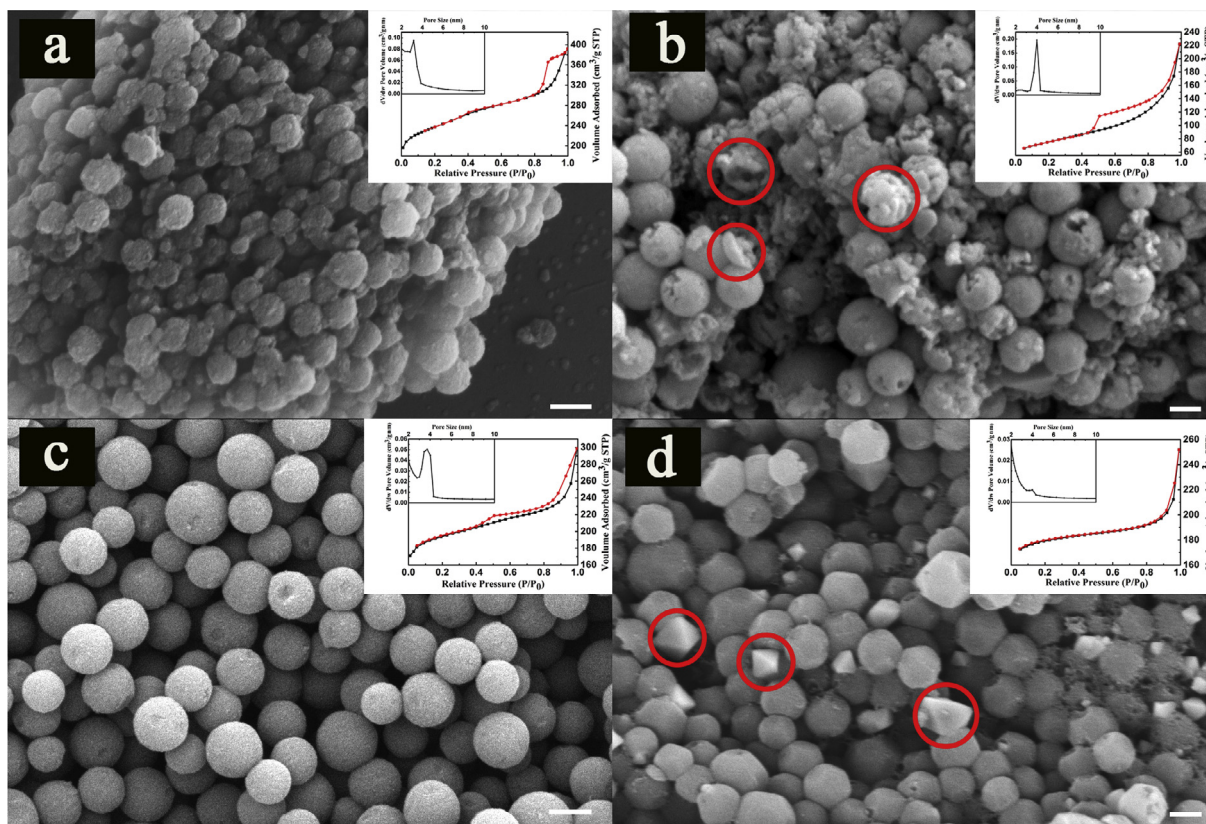


Fig. 1. SEM images, corresponding N_2 isotherms and PSD curves (inset) of the samples (a) MCNs, (b) MMCNs-1, (c) MMCNs-2 and (d) MMCNs-3. All the scale bars are 200 nm.

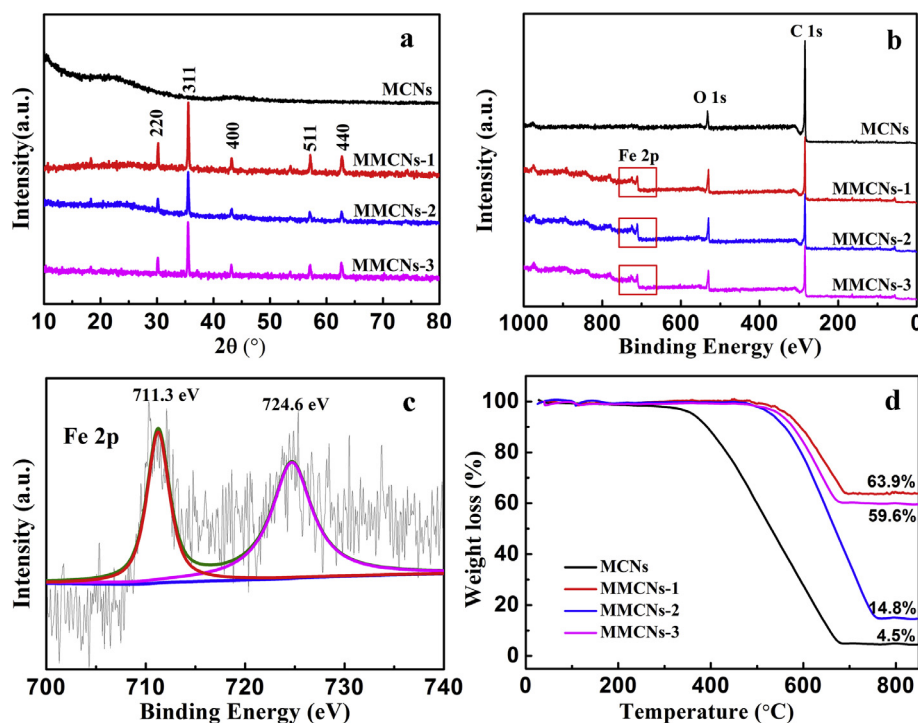


Fig. 2. The XRD (a), XPS (b), TG (d) of samples and XPS spectra of Fe 2p (c) for MMCNs-2.

nanoparticles are incorporated into mesoporous channels derived from removing F127, thus partially occupying or even fully blocking the mesopores. The MCNs (Fig. 1a) exhibit small particle size but adhere to each other. The serious particle aggregation is caused

by the linear framework of polymeric RF in acid aqueous solution, which would exhibit thermal instability. The MMCNs-1 (Fig. 1b) is slightly broken, this may be that the high content of Fe^{3+} consumes much carbon during calcination. For the MMCNs-3 (Fig. 1d),

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