SYNTHESIS OF HEMATITE-SILICA NANOCOMPOSITE HOLLOW SPHERES AND ITS CHARACTERIZATION

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Hematite (α-Fe_2O_3) particles with various morphologies have attracted much interest due to their technological applications including pigments, gas sensors, catalysts, and optical devices. The shape and size controlled α-Fe_2O_3 nanoparticles reveal a strong dependence of the magnetic properties on the nanostructure of the particles. We have developed a simple process to prepare the magnetic composite hollow spheres covered with nano-cluster [1]. As a continuous interest in magnetic hollow spheres, we report the preparation and magnetic behaviour of hollow α-Fe_2O_3/SiO_2 composite spheres with various sizes of α-Fe_2O_3 nanoparticles.

We have used functional polymer poly(St-co-MAA) as a core template to prepare hollow α-Fe_2O_3/SiO_2 spheres. The poly(St-co-MAA)/α-Fe_2O_3/SiO_2 core-shell sphere was prepared by the chemical co-precipitation and followed by a sol-gel method. To construct hollow α-Fe_2O_3/SiO_2 composite spheres, we took away the polymer core from the core-shell spheres using the THF to wash it and calcined in air at 450 °C for 4 hrs. To obtain α-Fe_2O_3 nanoparticles with different crystallite sizes, the hollow spheres were further calcined at 600°C for various times.

Fourier transform infrared spectra were used to characterize all samples. After calcination at 450°C, the characteristic peaks of the poly(St-co-MAA) disappear in the spectrum of hollow α-Fe_2O_3/SiO_2 spheres indicating that polymer cores have been completely removed from the hollow spheres. In addition, the content of α-Fe_2O_3 of the hollow α-Fe_2O_3/SiO_2 spheres is about 78 wt. % determined by the TGA. Fig. 1 shows the X-ray diffraction (XRD) patterns of the poly(St-co-MAA)/α-Fe_2O_3/SiO_2 core-shell spheres and hollow α-Fe_2O_3/SiO_2 spheres. A unique phase was identified as a corundum crystal structure for samples calcined at temperatures $T \geq 450$ °C. The mean crystallite size d of the coated α-Fe_2O_3 nanoparticles in hollow spheres is in the range of $2.2 \leq d \leq 5.2$ nm (inserted table of Fig. 3).

Fig. 2 presents the transmission electron microscope (TEM) images of poly(St-co-MAA) polymer spheres, poly(St-co-MAA)/α-Fe_2O_3 core-shell spheres, poly(St-co-MAA)/α-Fe_2O_3/SiO_2 core-shell spheres, and α-Fe_2O_3/SiO_2 hollow spheres. The poly(St-co-MAA) sphere has a diameter of 300 nm. Base on the results of XRD and TEM [Fig. 2 (b)~(d)], we think that the α-Fe_2O_3 nanoparticles coated on the surface of polymer is encaged in the nano-domain constructed by the SiO_2, which is like a nano-capsule, and the Fe_2O_3 particles is constrained in the nano-environment during high temperature treatment.

The hysteresis loops of the hollow α-Fe_2O_3/SiO_2 spheres display a superparamagnetic behavior at room temperature (Fig. 3). It is interesting that the hollow α-Fe_2O_3/SiO_2 spheres is a superparamagnet even though it was calcined at 600°C for 24 hrs. This result is consistent with the TEM and XRD that the Fe_2O_3 particles are not easily to grow fast in the nano-environment constructed by the SiO_2.

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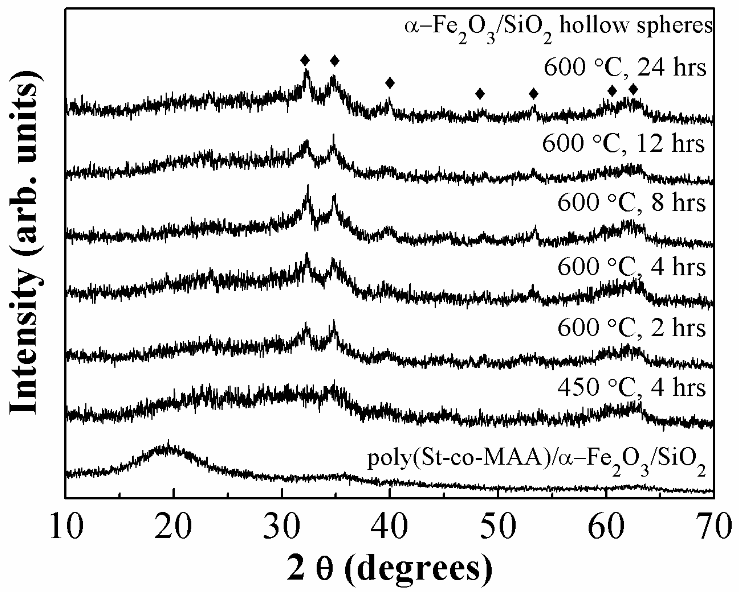

Fig. 1 XRD patterns of the poly(St-co-MAA)/α-Fe_2O_3/SiO_2 spheres and hollow α-Fe_2O_3/SiO_2 spheres.

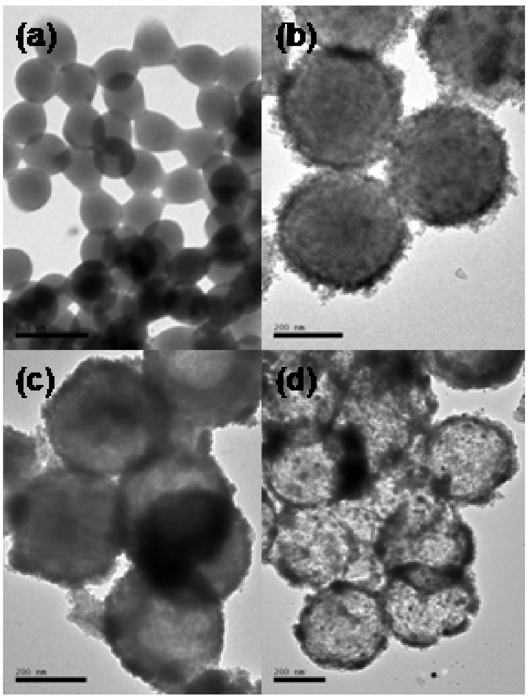

Fig. 2 TEM images of poly(St-co-MAA) spheres, poly(St-co-MAA)/α-Fe_2O_3 core-shell spheres, poly(St-co-MAA)/α-Fe_2O_3/SiO_2 core-shell spheres, and α-Fe_2O_3/SiO_2 hollow spheres (calcined at 450 °C).

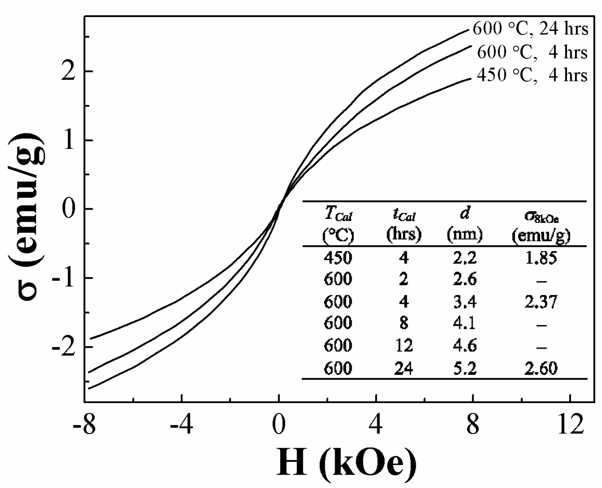

Fig. 3 Room temperature hysteresis loops of α-Fe_2O_3/SiO_2 hollow spheres prepared at 450 °C and 600 °C. The inserted table displays the calcined temperatures (T_{Cal}), calcined times (t_{Cal}), crystallite sizes (d), and magnetization (σ_{8kOe}) measured at 8 kOe.