SYNTHESIS AND CHARACTERIZATION OF CARBONIZED NANOMATERIALS BASED ON METAL-ORGANIC FRAMEWORK MIL-100(Fe)

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A series of carbonized metal–organic frameworks (MOFs) nanocomposite particles (NCPs) were synthesized by heat treatments MIL-100(Fe) at temperatures T_A between 300 °C and 800 °C in an argon gas. The MIL-100(Fe) were prepared by a hydrothermal synthesis method involves using $Fe(NO_3)_3 \cdot 9H_2O$ and 1,3,5-Benzenetricarboxylic acid well dissolved in the deionized water in a Teflon lined steel autoclave. XRD spectra show that the MIL-100(Fe) annealed at T_A between 400 °C and 600 °C contains pure cubic spinel (CS) phases. For $T_A \geq 700$ °C, the XRD patterns are dominated by the body-centered cubic (BCC) structural phase. However, a minor CS phase was detected at T_A =700 °C. The mean crystallite size of the CS and BCC phases in NCPs varied from 14.0 to 17.2 nm and from 42.1 to 44.3 nm, respectively. Raman spectra indicated the encapsulated carbon shells around the NCPs have the G-band and D-band centered at about 1580 cm^{-1} and 1330 cm^{-1}, respectively. Both linewidth of the G-band and D-band decreases with the annealing temperature. Magnetic measurements show that the MIL-100(Fe) and sample annealed at 300 °C are pure paramagnets while the samples annealed at $T_A \geq 400$ °C exhibits ferromagnetic behaviors. The saturation magnetization increases with increasing mean crystallite size for both CS and BCC structural phases. Magnetic force measurements were carried out as a function of temperature, with a TGA using a small permanent magnet, to determine the magnetic ordering temperature T_M of NCPs. We found two magnetic ordering temperatures T_{M1}=572 °C and T_{M2}=770 °C in the recorded curve. Both T_{M1} and T_{M2} correspond to the Curie temperature of cubic spinel iron oxides and α-Fe, respectively. Moreover, the heating curve shows a maximum T_H =721 °C just below the magnetic ordering temperatures T_{M2}. This thermomagnetic behaviour can be attributed to the Hopkinson effect which is governed by the temperature dependence of both the spontaneous magnetization and magnetic anisotropy of iron nanoparticles in NCPs.

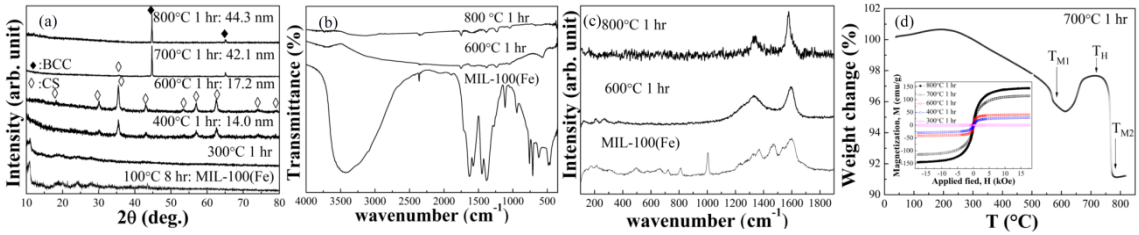

Figure 1 (a) XRD patterns of MIL-100(Fe), and MIL-100(Fe) carbonized at various temperatures; selected FT-IR (b) and Raman (c) spectra of MIL-100(Fe), and MIL-100(Fe) carbonized at 400 °C and 600 °C; (d) TGA curve of MIL-100(Fe) carbonized at 700 C recorded in an applied field & room temperature hysteresis loops of samples (inset).