Supplementary Material (ESI) for The Analyst

Optical sensor for the visual detection of mercury using mesoporous silica anchoring porphyrin moiety

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Supplementary Material

1. Characterization of SBA-15 and MFMS

The XRD pattern of the MFMS sample (Fig. 1) exhibit a well-resolved diffraction pattern corresponding to hexagonal structure with a prominent peak at 0.9^0 and two peaks at 1.65^0 and 1.85^0, as reported in the literature.[24] The TEM image of MFMS sample (Fig. 2) shows a typical, well-defined, hexagonal symmetry (P_{6mm}) viewed along the channel direction and confirms that hexagonal structure was retained even after functionalization of TMAC with silanol groups through covalent bonds. Nitrogen adsorption isotherms of both SBA-15 and MFMS samples were similar to those reported earlier.[25] The nitrogen adsorption-desorption isotherms (Fig. 3) were found to be of Type IV in nature as per the IUPAC classification and exhibits a H_1 hysteresis loop which is typical of mesoporous solids[25] with a sharp inflection at a relative pressure value of about 0.72 for SBA-15 and about 0.62 for MFMS. The mesopore diameter of SBA-15 was found to be 7.1 nm but it is reduced to 5.4 nm after grafting with TMAC (Fig. 3, inset). Similarly, the surface areas of both SBA-15 and MFMS samples were found to be 897 and 620 $m^2 g^{-1}$, respectively.

The reduced pore volume and surface area of MFMS was attributed to the presence of a certain amount of organic tethering inside the pores.

This was further confirmed by the thermo gravimetric analysis (TGA) of MFMS samples, which shows that about 19% of organic material and was stable upto 380°C. Furthermore, the FTIR spectra of siliceous and MFMS materials confirmed the presence of organic moieties in MFMS (Fig. 4). The appearance of a broad band around 3400 cm^{-1} indicates the presence of Si-OH asymmetric stretching. However, unlike siliceous SBA-15, MFMS material showed new bands at 2960 and 1400 cm^{-1} corresponding to aliphatic C-H bond and the stronger C-N bond, respectively. The bands at 1100 and 960 cm^{-1} were assigned to Si-O-Si and Si-OH stretching vibrations, respectively. The XPS analysis was performed over MFMS, TPPS-MFMS, and Hg-TPPS-MFMS materials to measure the relative concentration of C, O, N, Cl, Si, and Hg content. The XPS data indicate the presence of 'N' and 'Cl' in MFMS and 'Hg' along with 'N' in Hg-TPPS-MFMS materials confirming that Hg^{2+} effectively complexes with TPPS in MFMS material.

The density of the surface silanol groups attached to the SBA-15 was calculated[26] using thermogravimetry as 30.897 nm^{-2}, which is very high compared to the silica gel.[26] It is an advantage of this material for the reaction with TMAC and subsequently its high sensitivity for the detection of metal ions through chromophore.

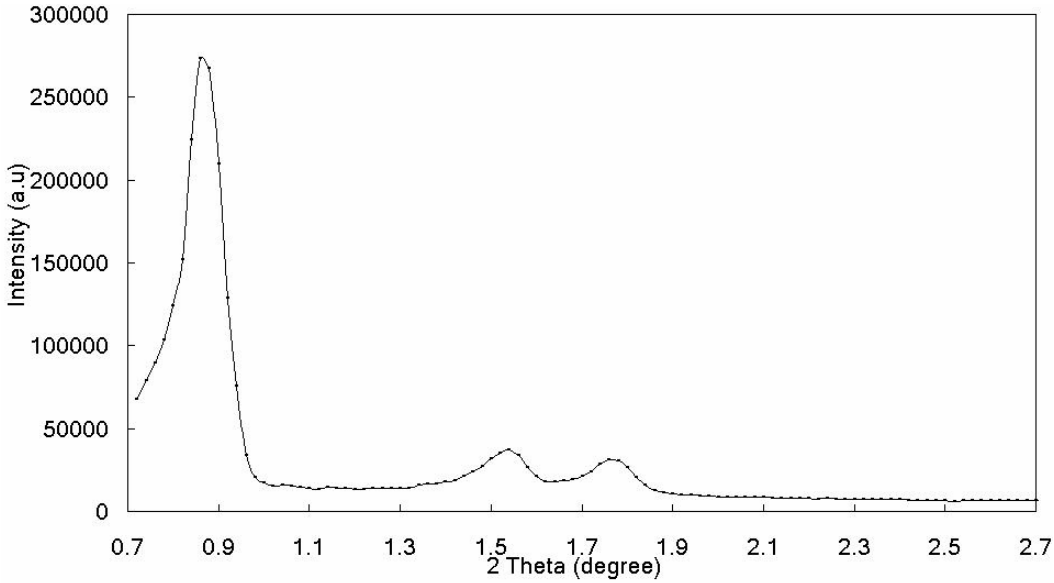

Fig. 1 XRD pattern of MFMS.

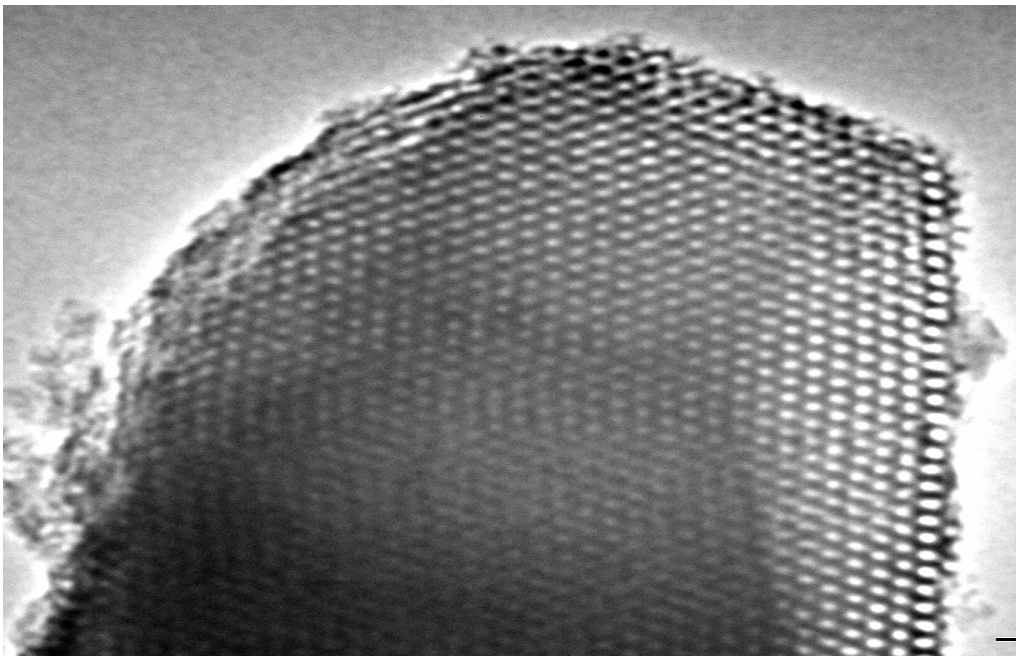

Fig. 2 TEM image of MFMS viewed along the channel direction.

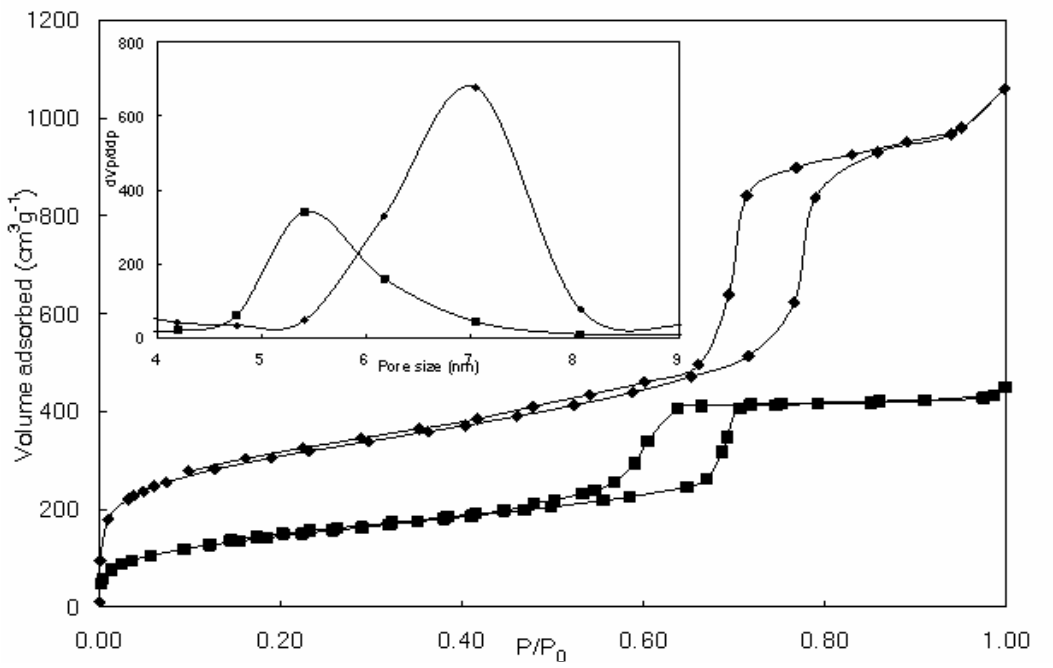

Fig. 3 Nitrogen adsorption and desorption isotherms of SBA-15 (◆) and MFMS (■), respectively. Inset: Pore size distribution evaluated by DH method.

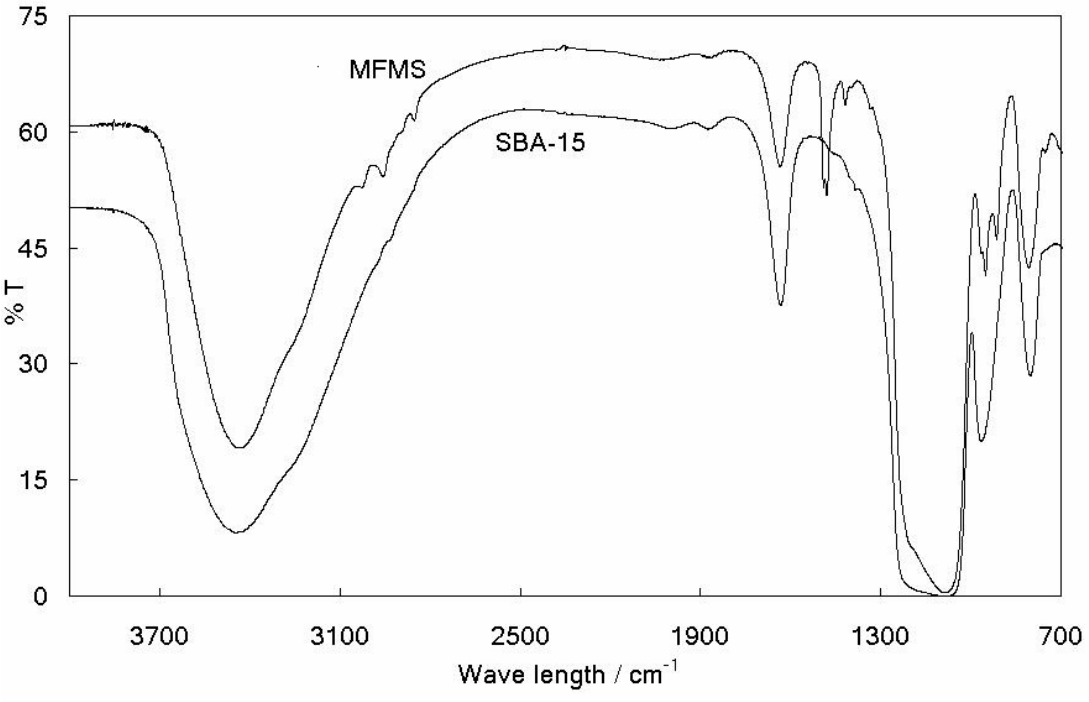

Fig. 4 IR spectra of SBA-15 and MFMS material.