## Abstract:

Fe-FSM-16 materials with different Si/Fe ratios (Si/Fe = 100, 50, 10) have been synthesized by intercalating kanemite using cetyltrimethylammonium bromide (CTMABr) as the intercalating agent and iron nitrate as the iron source, and characterized by several spectroscopic techniques. Electrons spin resonance and Mo"ssbauer spectroscopies, along with electron microscopy and X-ray diffraction, allowed differentiation of several iron species. These species correspond to (1) hematite particles, (2) very small "isolated" or oligomeric FeIII species possibly incorporated in the mesoporous silica wall, and (3) FeIII oxide clusters either isolated or agglomerated, forming "rafts" at the surface of the silica and exhibiting ferromagnetic ordering. Because of their agglomeration, these clusters appear with a two-peak size distribution, with one peak corresponding to the isolated clusters formed in the mesopores and still embedded in them and the other corresponding to the agglomerates spread on the surface of the mesoporous silica particles.