Experimental Report

Proposal: **1-04-62** **Council:** 10/2011

Title: SANS investigations on spray hydrolyzed mesoporous titania grains andits composites

This proposal is a new proposal

Researh Area: Materials

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Samples: TiO2

Instrument	Req. Days	All. Days	From	To
D11	1	1	30/05/2013	31/05/2013

Abstract:

Titania is an important material because of its significant role in environmental purification, given its photocatalytic properties. Spray hydrolysis is facile technique to synthesize such materials on industrial scale. Recently various morphologies of the spray hydrolyzed titania grains have been obtained by the control of physicochemical conditions. Titania composites are synthesized using this technique to achieve higher efficiency for solar spectrum. Titania microspheres will be doped with various elements such as N or C for improved photocatalytic application by tuning its band gap. The mesoscopic structure plays a very important role to get better photocatalytic activity. SANS/USANS is a unique tool to probe internal/overall structure of such grains. We propose to perform SANS (0.01 - 4 nm-1) and USANS experiments (0.0001 nm-1 -0.05 nm-1) covering a wide q range (~ 0.0001 to 4 nm-1)on spray-hydrolyzed porous titania grains and its composite prepared under various conditions. The closed and open porosity in these grains will be differentiated by performing contrast matching experiments.In addition,structure of the composites will be resolved by contrast match experiments.

SANS investigations on Co loaded mesoporous silica grains

Nano-composites, synthesized by evaporation induced self-assembly of and spray hydrolysis, possess hierarchical structures and find applications in various fields of technology that include catalysis, drug delivery etc. Morphology of the assembled grains and the interparticle correlation of the nanoparticles depend strongly on the physicochemical conditions during assembly process. Micro/Meso/ Macro pores may also be templated in such grains by selective removal of template materials after assembly. In order to understand, the internal structure of such grains SANS is a unique tool. The micrograph of mesoporous silica is shown in Fig. 1. Co element has been incorporated in the silica matrix to enhance the catalytic properties. Our aim in this work was to correlate the structure of the matrix with the catalytic performance of the material.

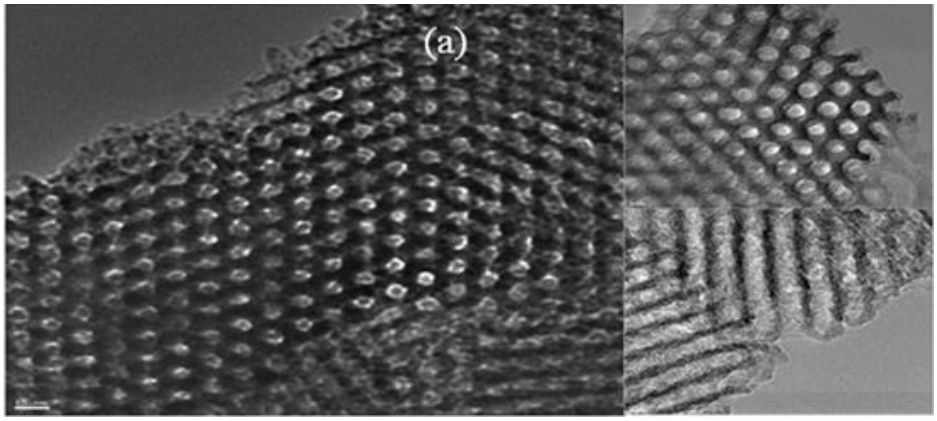

Fig. 1 TEM micrograph of the Cobalt loaded mesoporous silica is depicted

SANS experiments were carried out using D11. In order to access wide wave-vector transfer, sample to detector distance has been chosen as 2 m, 5.0 m, 8.0 m and 39 m. The overall accessed q-range was 0.002 $Å^{-1}$ to 0.33 $Å^{-1}$. The powder samples were wrapped in Aluminum foil or were put in quartz cuvette for measurements. The contrast variation experiments have been performed on the composite grains.

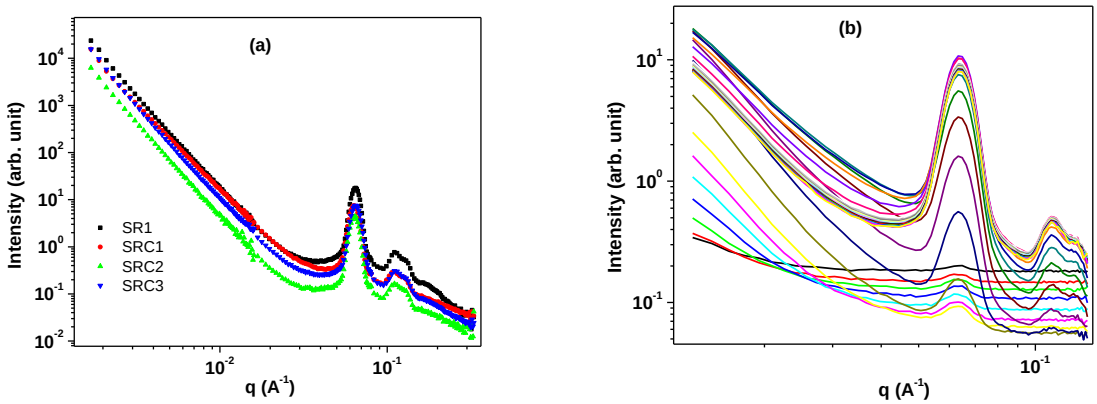

Fig. 2: *(a) Small-angle neutron scattering data from ordered porous silica composites. (b) The evolution of scattering profiles for the contrast matched composite at 140^{o}C.*

Diffraction intensity analysis of small-angle scattering measurements of dry catalysts confirms the ordered mesopore structure. Fig. 1(a) shows the scattering curve over the 0.0002-0.2 Å^{-1} range due to the coherent scattering from the hexagonal array of primary mesopores results in the (10), (11) and (20) Bragg diffraction peaks at 0.06, 0.104 and 0.13 Å^{-1} respectively for the HMS-X and Co-HMS-X catalysts [1-2]. The intensities of the higher order (11) and (20) diffraction peaks show that the material has highly ordered structure good long-range order. Contrast matching-small angle neutron scattering (CM-SANS) measurements have been carried out to probe the accessibility of the microspores using mixture of H_2O and D_2O. Typical survey scans are shown in Fig. 2(b). Here, diffraction intensity decreased below detection limit for the (10) peak at the contrast matching point and at the contrast match point, diffraction was eliminated completely, which implies that any micropores are larger than the water molecules and are completely accessible [1]. Thus, all the pores in the present probed length scale are open in nature.

References:

1. R. A. Pollock, B. R. Walsh, J. Fry, I. T. Ghampson, Y. B. Melnichenko, H. Kaiser, R. Pynn, W. J. DeSisto, M. C. Wheeler, B. G. Frederick, Chem. Mater. 2011, 23, 3828-3840
2. R. A. Pollock, G. Y. Gor, B. R. Walsh, J. Fry, I. T. Ghampson, Y. B. Melnichenko, H. Kaiser, W. J. DeSisto, M. C. Wheeler, B. G. Frederick, J. Phys. Chem. C 2012, 116, 22802-22814
3. S. Rahman, J. Bahadur, C. Santra, R. Kumar , A. Sultana, R. Schweins, D. Sen, , S. Maity, S. Mazumdar and B. Chowdhury 2014 (manuscript submitted).