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A Simple Method for the Synthesis and Characterization of Zn incorporated Thermally stable Hexagonal Mesoporous Silica based Molecular sieves

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Abstract: The discovery of periodic mesoporous molecular sieves attracts significant attention from a fundamental as well as applied perspective. The objective of this exertion is to synthesize the large pore Zn incorporated mesoporous molecular sieves by various mole ratios. The catalysts were prepared by simple sol-gel method. The obtained synthesized material was characterized by various spectroscopic techniques. Crystalline nature of the material was confirmed by low angle X- ray diffraction method. Nitrogen adsorption was used to determine the specific surface area, pore volume, pore diameter and pore wall thickness of the material. The weight loss and thermal decomposition of the material were analyzed by thermo gravimetric method. The Fourier Transform Infrared spectrum was taken for confirming the bonding characteristics of hexagonal mesoporous material in its various pH medium. The surface morphology of the material was confirmed by Scanning Electron Micrograph and Transmission Electron Micrograph. The results confirmed the formation of hexagonal mesoporous molecular sieves with high thermal stability (1200^{o}C) and large porosity (8.1).
Key words: mesoporous material, template, hexagonal, thermal stability, large pore.

Introduction and Experimental

Microporous and mesoporous solids have found great utility for applications in catalysis; separations, coatings and chemical sensing etc[1]. Recent attention has focused on mesoporous materials with larger pores and high thermal stability. We believe that we have developed a simple method to obtain highly reactive and thermally stable mesoporous Zn incorporated silica based molecular sieves from inexpensive silica source (Na_2SiO_3) and hydrocarbon template. The incorporation of different elements within the silica framework has been implemented in order to increase the acidity, ion exchange capacity and specific catalytic activity.

Result and Discussion

XRD patterns for different calcined samples are shown in Figure 1(A). A sharp diffraction peak (100) appeared around 2θ is equal to 1.1 in the calcined Zn incorporated sample and it is interpreted that the high

ordering and thermal stability of the mesoporous material[2]. The calcined material with low metal content exhibits weak signals between 2.3° and 2.8° due to the existence of (110) and (200) planes.

FT-IR spectra of the calcined sample (mole ratio 8) at various initial pH values are shown in Figure 1B. The difference in the initial synthesis pH value led to different spectral data suggesting the initial synthesis pH played an important role in the formation of new material. In the present study pH 10 has been chosen as the optimum synthesis gel pH for the preparation of novel mesoporous material. The lower pH (9-1) of synthesized gel revealed the deformation of asymmetric stretching of tetrahedral Si-O near 1093 cm^{-1} and higher pH (12-14) of the synthesized gel revealed low degree of order[3].

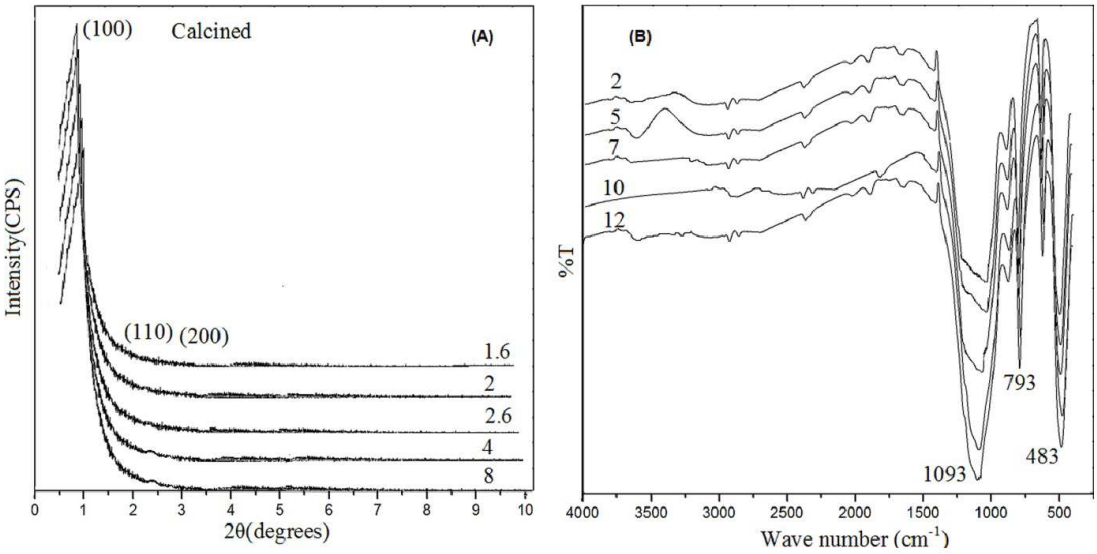

Figure 1: (A) XRD patterns of calcined sample at various mole ratios; **(B)** Effect of initial pH value of calcined sample (mole ratio 8)

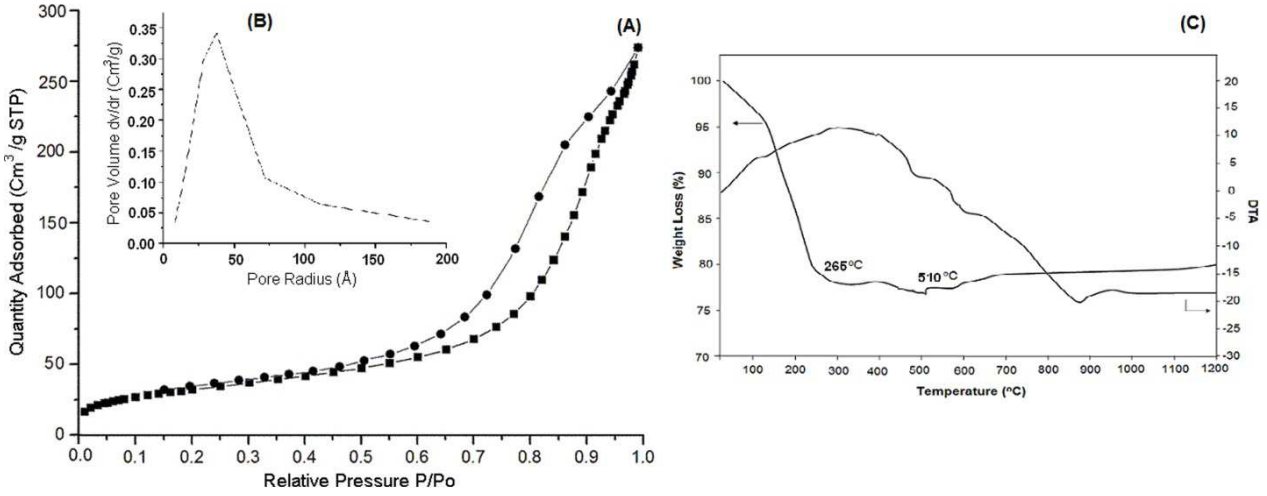

Figure 2 : **(A)** Nitrogen adsorption and desorption isotherm of calcined sample (mole ratio 8); **(B)** The corresponding BJH pore size distribution curve; **(C)** TGA and DTA analysis of as-synthesized material

A Nitrogen adsorption isotherm for the material under study is shown in Figure 2(A). The BET specific surface areas, total pore volumes and pore wall thickness are listed in Table - 1. N_2 adsorption at low relative pressure ($P/P_0 < 0.3$) was accounted for monolayer adsorption of N2 on the walls of the mesopores[4]. The P/P_0 of 0.05–0.95 was corresponding to capillary condensation within framework of textural porosity.

Table-1: Physicochemical parameters of Zn incorporated mesoporous molecular sieves

Si/Zn-mole ratio	d-spacing [a] (nm)	a_0 -Unit cell parameter[a] (nm)	BET-surface area[b] m^2/g	Pore size[b] (nm)	Pore volume[b] (cm^3/g)	Pore wall thickness[c] (nm)
8	8.03	9.27	98	8.1	0.35	1.17
4	8.17	9.44	95	8.3	0.33	1.14
2.6	8.49	9.80	92	8.6	0.32	1.20
2	8.65	9.99	85	8.8	0.29	1.19
1.6	8.83	10.19	78	9.1	0.27	1.09

[a] Values obtained from XRD studies.
[b] Values obtained from N2 adsorption-desorption results.
[c] Wall thickness = unit cell parameter - pore size.

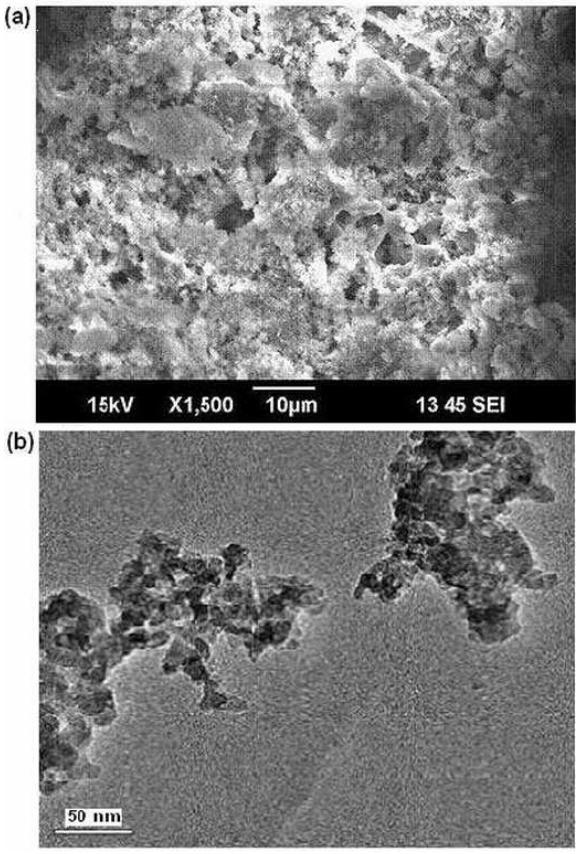

Figure 3 : (a) SEM image; **(b)** TEM image

Thermal stability of the as-synthesized sample was characterized by thermo gravimetric method. The weight loss from 100^{o}C to 265^{o}C was attributed to desorption of adsorbed water and the decomposition of template molecules[5]. The weight loss of 2.6% was recognized between 265–510^{o}C, which corresponds to the condensation of adjacent OH group. The additional increase of temperature reveals that there is no weight loss in the material which proves the synthesized material was stable up to 1200^{o}C; whereas the earlier reported mesoporous materials were stable up to 800^{o}C only. SEM and TEM images of the mesoporous hexagonal silica obtained from Si/Zn mole ratio 8 displayed in Figure 3. The SEM image of the sample (Figure 3a) shows a rod like hexagonal or spherical morphology and with the particle size ranges from 150 to 200 nm[6]. The morphology was further identified with the HRTEM observations shown in (Figure 3b).

It is concluded that this characterization technique confirms the formation of novel Zn incorporated mesoporous hexagonal silica based molecular sieves with large pore and high thermal stability by using hydrocarbon as a template.

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