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## NANO COMPOSITE CATALYTIC MATERIALS: SYNTHESIS AND CHARACTERIZATION

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### Abstract:

A novel precipitating agent hexa methyl tetramene is used for synthesis of mixed metal oxide nano composite catalysts. The catalysts synthesized were analysed by X-Ray diffraction, Surface area by Brunauer, Emmett and Teller technique (BET) and Transmission electron microgram (TEM).

**Keywords:** Brunauer, Emmett and Teller technique and Transmission electron microgram.

### 1. Introduction

In the recent times, considerable developments in the preparation of well ordered mesoporous materials have triggered research efforts in catalysis, catalysis supports and adsorbents because of their high-specific surface areas, large specific pore volumes, and narrow pore size distribution<sup>1(a-g)</sup>. Crystalline nanomaterial's consisting of complex metal or metal oxides are generally known as nanocomposites. Metal or metal oxide nanoparticles or their nanocomposites are usually synthesized by electrochemical deposition or chemical deposition methods followed by decomposition or reduction in the confined channels of host materials such as carbon nanotubes<sup>2(a-b)</sup>, anodic aluminum membranes<sup>3</sup>, and micro porous or mesoporous molecular sieves<sup>4(a-c)</sup>.

In the traditional strategy, such as wetness impregnation or ion exchange, metal salt diffuse easily to the outer surface of the host silica to form large metal aggregates during the reduction or thermal treatment process.<sup>5(a-c)</sup> At this juncture, formation of metastable solid solution<sup>6(a-b)</sup> or introduction of second phase is an indispensable requirement to prevent the particle growth or sintering of nanoparticles. By using the second metal and without using harmful organic precursors, solvents, templates and capping agents, Mixed metal oxide Nano composite catalysts of SnO<sub>2</sub> with CeO<sub>2</sub> and ZrO<sub>2</sub> are prepared by a novel technique using hex methyl tetramene as a precipitating agent.

## 2. Experimental procedure:

### 2.1 Preparation of Mixed metal oxide Nano composite of SnO<sub>2</sub> with CeO<sub>2</sub> and ZrO<sub>2</sub>

CeO<sub>2</sub> and ZrO<sub>2</sub> nano composites were prepared by using hexamethylene tetramene as precipitation agent. Hydrolysis of cerium salt as well as tin salts was carried out by adopting thermal treatments. Mixed metal oxide Nano composite of SnO<sub>2</sub> with CeO<sub>2</sub> and ZrO<sub>2</sub> materials were analyzed by X-ray diffraction, surface area by BET and Transmission electron microgram (TEM)

### 2.2. Characterization by X-Ray Diffraction:

XRD patterns of nanocomposites were recorded by a Rigaku Miniflex X-ray diffractometer (M/S. Rigaku Corporation, Japan) using Ni filtered Cu K<sub>α</sub> radiation with scan speed 2°min<sup>-1</sup> and 2θ range from 10-80°. The low angle XRD patterns were obtained from Altima –IV Rigaku X-ray diffractometer (M/S. Rigaku Corporation, Japan) using Ni filtered Cu K<sub>α</sub> radiation with scan 1°min<sup>-1</sup> and 2θ range from 0.7-5°.

### 2.3. Characterization by BET Surface Area

Surface area was determined by BET single point method, single point measurements are accomplished using a 30% N<sub>2</sub> /70% He gas mixture. The sample was pre-heated to remove all moisture in flow of pure He (99.99 %) at 473 K for 2 h. After the completion of pretreatment N<sub>2</sub> through the sample tube, which is placed in a liquid N<sub>2</sub> bath (77 K).

### 2.4. Characterization by Transmission electron microscopy

The nanocomposite samples prepared in this project work were characterized by transmission electron microscopy (TEM) (M/s. Phillips, Netherlands, Model: Tecnai Feil2, capacity: 120 KV). The finally powdered nanocomposite samples were sonicated in methanol for about 15 min and placed on sample holders (made of gold) and conducted the TEM analysis.

## 3. Results and discussion:

### 3.1 Characterization of mixed metal oxide nano composite materials by XRD

The sharp diffraction reflections of the XRD pattern of SnO<sub>2</sub>CeO<sub>2</sub> centered at around 2θ = 26.7 (110), 33.9 (101), 37.9 (200), 51.8 (211) 54.8 (220) and 62.0(310) are attributed to tetragonal rutile crystalline phase of SnO<sub>2</sub> (Fig. 8) with a reference pattern (JCPDS 41-1445). The narrow XRD peaks of SnO<sub>2</sub> (SnO<sub>2</sub>CeO<sub>2</sub>) catalyst demonstrated the well-crystallized particles. The diffraction lines of pure cerium oxide (SnO<sub>2</sub>CeO<sub>2</sub>) are at around 2θ = 28.4 (111), 33.0 (200), 47.5 (220), 56.3° (311) correspond to cubic fluorite (Fm $\bar{3}$ m, 225) structured CeO<sub>2</sub> (JCPDS 43-1002). The diffraction peaks of SnO<sub>2</sub>CeO<sub>2</sub> are matches well

with the pure CeO<sub>2</sub> and no other cerium oxide phases are discerned. Although there is 10% SnO<sub>2</sub> in the Sn Ce<sub>9</sub> sample, no diffraction peaks due to metallic Sn or tin oxide are detected, which is an indication of solid solution formation and furthermore, the broadened diffraction peaks of Sn<sub>1</sub>Ce<sub>9</sub> represent the smaller crystallites. The Bragg's XRD pattern of Sn<sub>5</sub>Ce<sub>5</sub> sample exhibits both the phases of CeO<sub>2</sub> and SnO<sub>2</sub>. This XRD analysis clearly indicates the phase segregation. In other words solid solution formation occurs only at a certain composition. When the composition of SnO<sub>2</sub> and CeO<sub>2</sub> reaches to 50% certain amount of SnO<sub>2</sub> phase is segregated. At 90% SnO<sub>2</sub> (CeO<sub>2</sub>SnO<sub>2</sub>) composition level SnO<sub>2</sub> is a dominant phase. From the XRD analysis it is clear that the mixed oxide nanoparticle of SnO<sub>2</sub> and CeO<sub>2</sub> at all the composition levels the size of the particle is smaller than that of isolated oxides.

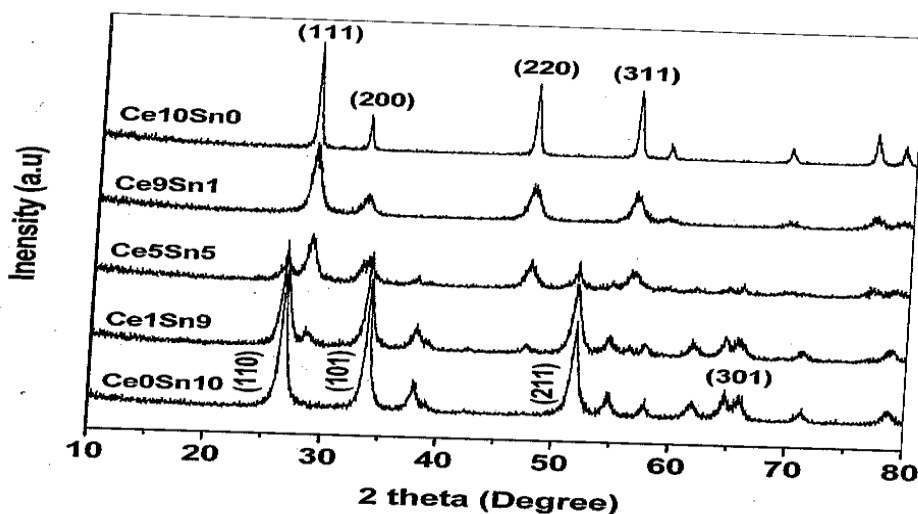


Fig-1: XRD pattern of SnO<sub>2</sub> and CeO<sub>2</sub> system.

The XRD patterns of SnO<sub>2</sub>-ZrO<sub>2</sub> binary mixed metal oxide nanocatalysts and isolated SnO<sub>2</sub> (Sn<sub>10</sub>Zr<sub>0</sub>) and ZrO<sub>2</sub> (Sn<sub>0</sub>Zr<sub>10</sub>) are depicted in Fig. 10. Sn<sub>0</sub>Zr<sub>10</sub> nanocatalyst exhibited sharp diffraction peaks at  $2\theta = 24.1(110)$ ,  $28.1(11-1)$ ,  $31.3(111)$ ,  $34.1(200)$  and  $50.2(022)$ , which are well consistent with the monoclinic phase of ZrO<sub>2</sub> according to the ICDD No 37-1484. No other crystalline phases of ZrO<sub>2</sub> have been observed.

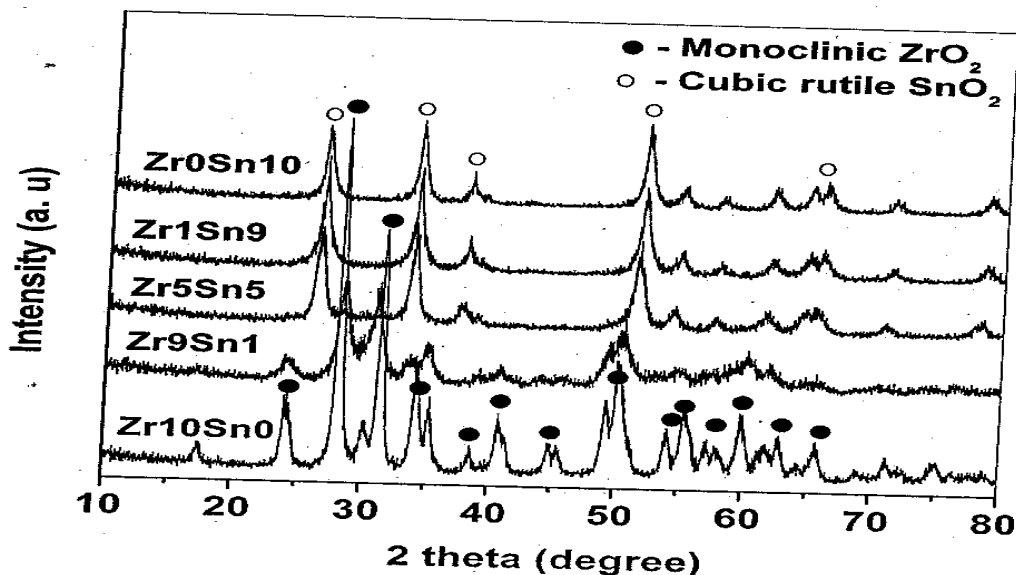


Fig-2: XRD pattern of SnO<sub>2</sub> and ZrO<sub>2</sub> system.

3.2 Characterization of mixed metal oxide nano composite materials by TEM.

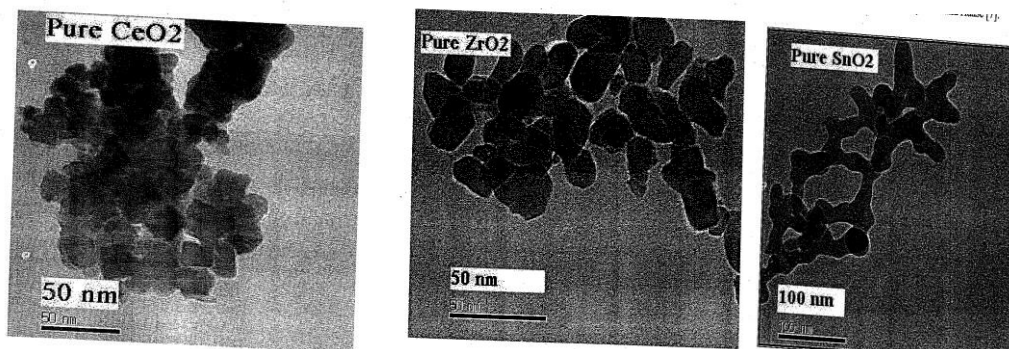


Fig-3: TEM images of Metal oxide.

3.3 Characterization of mixed metal oxide nano composite materials by BET surface area.

S.No	Nano Composite	BET Surface area (m <sup>2</sup> /g)
1	SnO <sub>2</sub>	12
2	ZrO <sub>2</sub>	20
3	CeO <sub>2</sub>	9
4	CeO <sub>2</sub> -SnO <sub>2</sub>	97
5	ZrO <sub>2</sub> -SnO <sub>2</sub>	152
6	SnO <sub>2</sub> -CeO <sub>2</sub> -ZrO <sub>2</sub>	165

#### 4. Conclusion:

Using the green synthetic approach (GSA) method other tin based nanocomposites such as SnO<sub>2</sub>-CeO<sub>2</sub> and SnO<sub>2</sub>-ZrO<sub>2</sub> were synthesized and were analyzed by X-ray diffraction, surface area by BET and Transmission electron microgram (TEM)

#### 5. References:

- 1 (a) A. sayari, *Chem. Mater.* 8 (1996) 1840, (b) A. Corma, *Chem. Rev.* 97 (1997) 2373, (c) J.Y. Ying, C.P. Mehnert, M.S. Wong, *Angew. Chem.Int.Ed.* 38 (1999) 56, (d) A. Hagfeldt, M. Gratzel, *Acc. Chem. Res.* 33 (2000) 269, (e) Y.Tao, H. Kanoh, L.Abrams, K.Kaneko, *Chem, Rev.* 106 (2006) 896 (f) J.A.Melero, R. Van Grieken, G. Morales, *Chem, Rev.* 106 (2006) 3790 (g) M. Valler-Reg, *Chem. Eur. J.* 12 (2006) 5934.
- 2 (a) D.Wang, R.A.Caruso, F.Caruso, *Chem. Mater.* 13 (2001) 364, (b) A.Imhof, G.J.Pine, *Adv.Mater.* 11 (1999) 311.
3. R.D.Muller, *Science* 286 (1999) 241.
4. (a) S.Shimuzu, H. Hamada, *Adv.Mater.* 1 (2000) 1332, (b) Y.Lee, J.S.Lee, Y.S. Park, K.B. Yoon, *Adv. Mater.* 13 (2001) 1259, (c) S. Mintova, T. Bein, *Adv. Mater.* 13 (2001) 1880.
- 5 (a) J.-L. Shi, Hua, L.-X Zhang, *J. Mater. Chem.* 14 (2004) 795, (b) K.Yamamoto, Y. Sunagawa, H. Takahashi, A. Muramatsu, *Chem. Commun.* (2005) 348, (c) P. Han, X. Wang, X Qiu, X. Ji, L Gao, *J. Mol. Catal. A: Chem.* 272 (2007) 136.
- 6 (a). E.R.Leite, A. p. Maciel, I. T. Weber, P. N. Liaboa-Filho, E. Longo, C. O. Paiva-Santos, A.V.C. Andrade, C. A. Pakoscimas, Y. Maniette and W.H. Shreiner, *Adv. Matter.* 14 (2002) 905.(b). E.R.Leite, I. T. Weber, E. Longo, J.A. Varela, *Adv. Mater.* 12 (2000) 965.
7. D.Zhao, Q.Hua, J.Feng, B.F.Chmelka, G.D.Stucky, *J.Am.Chem.Soc.* 120 (1998) 6024.

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