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Combustion Synthesis of Calcium Zirconate Ceramic Nano Powders - Their Structural and Morphological Studies

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ABSTRACT

The present paper gives a brief insight of preparation of calcium zirconate ($CaZrO_3$) dense ceramic nanopowders by simple solution combustion method using urea as a fuel. The prepared material was characterized by X-ray diffraction (XRD), field emission scanning electron microscopy (FESEM) and energy dispersive spectroscopy (EDX). XRD data clearly shows the crystallization of the CaO-ZrO_2 Nano Powders and the formation of calcium zirconate solid solutions in the wt % ranging from 2-10 wt%. From the SEM data of the material (with different concentration of CaO) it was evident that, in all the samples the particles are agglomerated and showed irregular morphology.

Keywords: S*olution combustion synthesis, Ceramics and Nano powders.*

INTRODUCTION

In the recent years globally extensive attention has been given to the ceramic materials for various applications. CaO and ZrO_2 were the materials explored for their thermal resistance [1], electrical resistance [2], mechanical strength [3], durability and chemical resistance [4], catalytic properties [5]. Calcium zirconate is the most stable combination of all the quasi binary system, also exhibit narrow range of homogeneity. Due to the excellent properties of these composites, they are used in solid oxide fuel

cells [6], dense ceramics and bulk catalysts. These composite nanomaterials can be prepared by various methods like co-precipitation [7], combustion [8] and hydrothermal [9], sol-gel [10] etc., out of which solution combustion process is one of the promising and rapid method for the synthesis of nanomaterials. As many methods result in the formation of homogeneous solid solutions and heterogeneous solid solutions, this method not only provides easy way of synthesis, but also generates heterogeneous solid solution.

MATERIALS AND METHODS

XRD measurements were done using Rigaku smart lab, EDX and SEM images were recorded using Zeiss scanning electron microscope, Calcium zirconate ceramic nano powders were prepared by mixing stoichiometric coefficients of calcium nitrate, zirconium oxy nitrate and urea, further homogenized on a magnetic stirrer to obtain a clear solution, this solution obtained was subjected to combustion in a preheated muffle furnace maintained at 400°C. The combustion reaction facilitated with the evolution of gases and leaving behind voluminous white powder. The obtained powder was crushed to fine and again calcinated at 800°C, to obtain phase pure ceramic powders a schematic representation of the synthesis process is shown in the Figure 1.

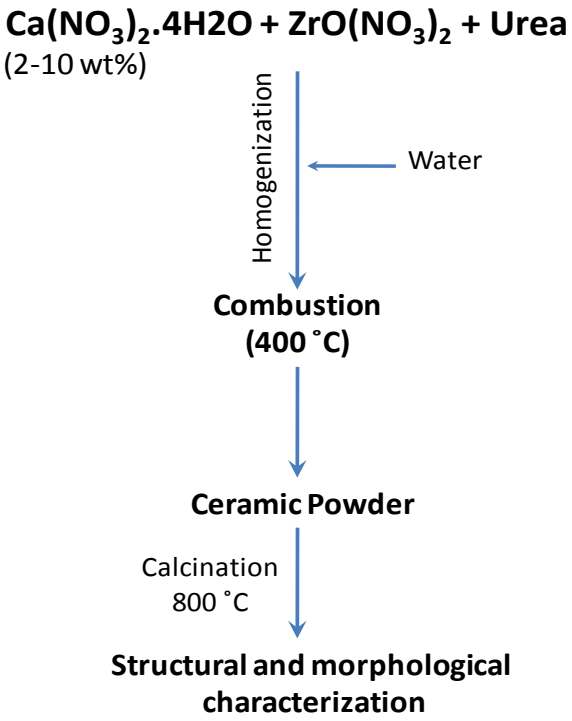

Figure 1: Schematic representation of synthesis protocol.

RESULTS AND DISCUSSION

Structural characterization of preapared calcium zirconate ceramic nano powders

The X-ray patterns were recorded for all the samples with Rigaku smart lab. Phase formation and crystal structure of the calcium zirconate nano powders calcinated at 800°C was studied by XRD analysis with Cu-Kα radiation source having a wavelength of 1.54056Å, the obtained pattern was shown in Figure 1. From the figure it is evident that the catalyst has distinct XRD pattern with intensity peaks at 2θ = 30.11, 35.10, 50.26, 59.88, 62.83, 74.25, 81.95 and 84.78, indicating cubic structure of calcium zirconate powders, which are in good agreement of crystal data obtained from PDF card number 3-640.

The average particle size was calculated using Scherrer equation:

$$D=K\lambda/\beta\cos\theta$$

Where λ is the wavelength of X-rays, K is crystallite shape factor which was assumed to be 0.94, θ is the diffraction angle and β is the corrected full width at half maximum, Table 1 provides the detailed calculation of crystallite size using Scherrer formula and the average crystallite size was found to be 8-21 nm. A graph of 4sinθ and βcosθ was plotted along x and y axis respectively, the slope of the graph provides the strain in the lattice and y-intercept gives the crystallite size as per Williamson-Hall plots (W-H plots) and the crystallite size calculated to be in the range of 7.5-49 nm using $\beta\cos\theta = (k\lambda/D) + 4\varepsilon\sin\theta$ (Williamson hall method).

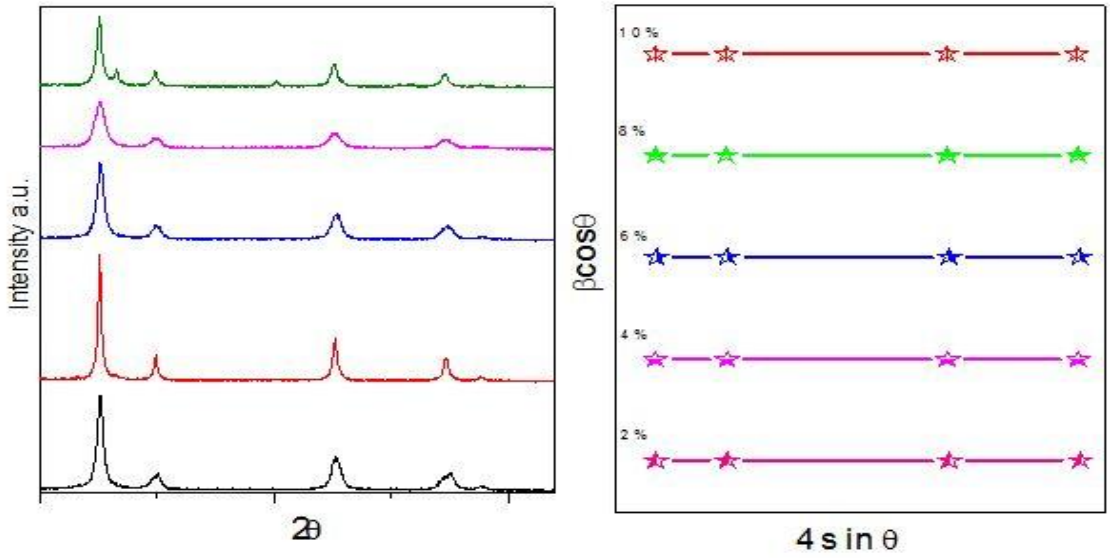

Figure 2: (a) XRD patterns of calcium zirconate (2-10 wt%) nano powders (b) Williamson-Hall plots of calcium zirconate nano powders (2-10 wt%).

Table 1: Calculation of crystallite size, strain and dislocation density from the XRD data

CaO doped (wt %)	Crystallite size			Dislocation density '1/D^2'	
	Scherrer method	W-H method	Strain 'ε' 10^4	Scherrer method	W-H method
2	10.57	47.9512053	29.02752	0.008951	0.000435
4	20.89	26.81715556	8.748621	0.002292	0.001391
6	10.26	11.78296501	7.409295	0.0095	0.007203
8	8.36	7.507135303	-6.56385	0.014308	0.017744
10	14.7	13.03444104	-7.99887	0.004628	0.005886

SEM and EDX analysis

SEM micrographs are recorded with ZEISS Field Emission Scanning Electron Microscope; the micrographs are as shown in the Figure 2 (a) to (e) for 2-10 wt % of calcium zirconates nano powders as mentioned in the Figure 2. The results reveals that the preparation of the calcium zirconate by the protocol mentioned in the experimental section and subsequent reaction conditions lead to the formation of irregular shaped agglomerated particles as clearly evident from the SEM data. The calcium zirconate has porous structure with high surface area with high agglomeration which is the specific nature when the materials are prepared through co-precipitation technique, however this can be avoided by adding suitable surfactants or structure modifiers. The porous nature may be arising due to the escape of water molecules from the lattice surface during the course of calcination at 400^{o}C. From the elemental analysis it is clearly evident that calcium and zirconium are present in the lattice, more over no significant peaks for calcium oxide in the XRD indicates high dispersion of calcium in the sea of zirconia lattice.

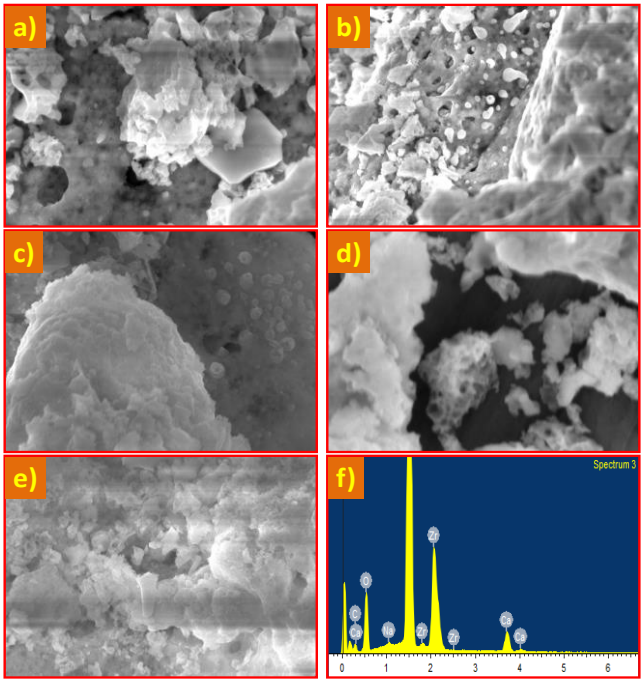

Figure 3: (a - e) FESEM images of calcium zirconate nanopowders (2-10 wt%) and (f) EDX spectrum of $CaZrO_3$ (8 wt%).

CONCLUSION

In conclusion calcium zirconate nano powders were prepared by simple solution combustion process. Phase formation and crystallization were confirmed by the XRD data, morphological features and elemental composition were studied by SEM and EDX analysis.

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