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Study of $ZnAl_2O_4$ Prepared by Co-precipitation Method

Shyam Sunder[1], Atul Kumar[2]
[1] Department of Applied Science, CDL State Institute of Engineering &Technology Panniwala Mota, Sirsa
[2] Department of Physics, Government Senior Secondary School, Bhuratwala, Sirsa, India

Abstract: Zinc aluminate is well known wide bandgap semiconductor with cubic spinel structure and transparent for wavelength greater than 320 nm. Therefore, $ZnAl_2O_4$ can be used for ultraviolet photoelectronic devices. Furthermore, spinel zinc aluminate is useful in many reactions as catalytic support. Moreover, zinc aluminate can be used as second phase in glaze layer of white ceramics to improve wear resistance and to preserve whiteness. In present study cubic spinel zinc aluminate nanoparticles have been synthesized from aqueous solution of $Zn(NO_3)_2.6H_2O$ (0.1 M) and $Al(NO_3)_2.9H_2O$ (0.2 M) using chemical coprecipitation technique. Ammonium hydroxide was used as precipitating agent and pH was maintained between 8 to 9. The precipitated slurry was filtered and washed several times with deionized double distilled water and dried at 110 ^{0}C. The fine powder was annealed at different temperatures from 600 ^{0}C to 900 ^{0}C for 4h in temperature controlled furnace. Structural characterization of annealed samples was carried out via X-ray Diffraction (XRD), and Fourier Transform Infrared spectroscopy (FTIR). XRD patterns reveal that zinc aluminate samples were cubic spinel nanoparticles and grain size determined by Debye-Scherrer formula is from 5 to 16 nm.

Keywords: Nanoparticles, Coprecipitation, Zinc aluminate, XRD, FTIR. **PACS:** 81.07.Wx, 81.07.Bc.

INTRODUCTION

The zinc aluminate has a spinel structure and found in nature as a mineral named gahnite. It is a typical example of compounds of general formula $(A)[B]O_4$, where A and B are divalent and trivalent ions respectively [1]. The decrease in the grain size of the zinc aluminate nanoparticles, is leading to many intresting and extraordinary electrical, mechanical and optical properties unknown in the bulk material. Due to these properties zinc aluminate can be used as ceramic material, wide bandgap semiconductor and in optic coating in aerospace applications. Furthermore zinc aluminate can be used as catalytic support for different reactions, such as paraffins dehydrogenation, saturated alcohols dehydration to olefins, methanol and heavy alcohols synthesis, since it has a high thermal stability, low acidity and a hydrophobic behavior [2, 3]. Coprecipitation is a simplest and inexpensive technique for preparing nanoparticles of mixed inorganic materials. In this paper, the preparation of nanosized zinc aluminate spinel by means of coprecipitation route using ammonia solution as chelating agent, starting from mixed metal nitrates [4-6]. Characterization of the nanopowder was carried out by using Powder X-ray diffractometer (XRD) and Fourier Transform Infrared Spectrophotometer (FTIR).

EXPERIMENTAL

All chemicals were purchased from Sigma Aldrich or Fluka and used without further purification.

Sample Preparation

Zinc aluminate nanoparticles were prepared in aqueous solution from nitrates of Zn and Al by coprecipitation method using ammonia as a precipitating agent. Stoichiometric amount of $Zn(NO_3)_2.6H_2O$ (0.1 M) and $Al(NO_3)_2.H_2O$ (0.2 M) solution has been prepared and mixed rigorously using magnetic stirrer. Then, the appropriate amount of aqueous ammonia solution (25% V/V) was added to the above mixed solution, and mixture was stirred until complete precipitation occurs at a pH between 8 and 9 and reaction temperature was maintained at 50 ^oC. The precipitate was filtered, washed with distilled water many times, and dried in air at 110 ^oC. The dry precipitate was heat treated at 600, 700, 800, 900 ^oC for 4h to obtain the $ZnAl_2O_4$ nanoparticles.

Characterization

The $ZnAl_2O_4$ nanoparticles were characterized by X-ray diffraction (XRD) and Fourier Transform Infrared (FTIR) spectroscopy. X-ray diffraction patterns were recorded at room temperature in a Rigaku Miniflex-II XRD using CuKα radiation (λ=1.5412 A), generated at 30 kV and a current of 15 mA in the range of 2θ from 10^0 to 80^0. Fourier Transform Infrared Spectra were recorded in a Perkin Elmer FTIR Spectrometer with range from 4000 to 400 cm^{-1} using the pellets of the sample with KBr.

RESULTS AND DISCUSSION

In coprecipitation technique, aqueous solution of required metal salts is coprecipitated using an appropriate precipitating agent. The precipitates so obtained are solid solution that contains the cations of the metal salt and require certain temperature for the reaction to occur. The process for the preparation of $ZnAl_2O_4$ from the precursor and precipitating agent is given below:

$$Zn(NO_3)_2 + 2Al(NO_3)_3 \xrightarrow[50\,^oC \quad 110^oC]{NH_4OH} Zn(OH)_2.2Al(OH)_2 \xrightarrow{\Delta} ZnAl_2O_4 + H_2O$$

1) XRD Studies:

The XRD spectra of the synthesized $ZnAl_2O_4$ nanopowder sample heated at 700 ^oC for 4h, in air is given in Fig. 1. The peaks appearing at 2ϴ= 31.44^o, 37.04^o, 45.10^o, 55.86^o, 59.56^o and 65.52^o. They can be indexed as (220), (311), (400), (422), (511) and (440) crystal planes of the cubic spinel crystalline structure of $ZnAl_2O_4$ respectively, in accordance with the standard JCPDS card of cubic spinel-type $ZnAl_2O_4$ (JCDPS card No. 05-0669).

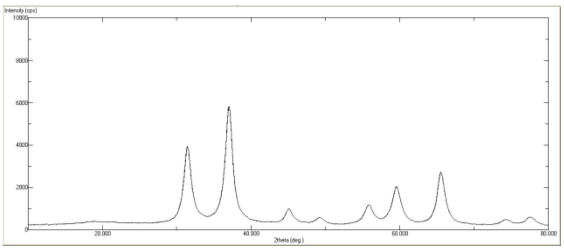

FIGURE 1. XRD pattern of $ZnAl_2O_4$ nanoparticles heat treated at 700 ^oC.

The prominent peak was observed at 37.04^o corresponding to (311) crystal plane. Full crystallization of the $ZnAl_2O_4$ was achieved at 700^{o}C. The XRD pattern shows that the prepared zinc aluminate sample is single phase and the peaks detected are of characteristic peaks of cubic spinel $ZnAl_2O_4$. The average particle size was calculated by Deby-Scherrer formula using XRD pattern, $d=(0.9\lambda)/(\beta\cos\theta)$, where d is the grain size, λ is the wavelength of the X-ray used, θ is the diffraction angle, β is the full width at half maxima of the peak. The average size so calculated corresponding to prominent peak (311) was 7 nm.

2) FTIR Studies:

The IR spectra of the prepared Zinc aluminate sample is shown in Fig. 2. Three bands observed between 900 to 400 cm^{-1} are related to inorganic network [9].

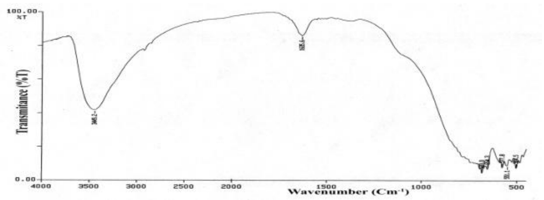

FIGURE 2. FTIR spectra of $ZnAl_2O_4$ nanoparticles heat treated at 700 ^oC.

Bands at 654 and 551 cm^{-1} are attributed to Al-O stretching and O-Al-O bending vibrations of AlO_6 group in spinel type $ZnAl_2O_4$ structure [7, 8], respectively. Band at 3449 cm^{-1} is related to the vibration of OH group bonded to the surface. Band observed around 1635 cm^{-1} is associated to the HOH due to the entrapped water content [10]. The further study is to be carried out and will be reported soon in journal.

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