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Effect of calcination on morphology and optical properties of AlO-CeO nanocomposites

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ABSTRACT

In the present investigation, main emphasis has been given to synthesize AlO-CeO nanocomposites by a Sol-Gel method based on polymeric network of polyvinyl alcohol (PVA). The idea behind sol-gel synthesis is to "dissolve" the compound in a liquid in order to bring it back as a solid in a controlled manner. In this method mixture solvent of 50:50 ethanol-water was used to dissolve aluminium nitrate, cerium nitrate and PVA. The mixture was heated to 80° C to form homogeneous gel. The hard gel was calcined at a temperature of 600° C for 4 hour and 8 hour and finally converted into nanocomposites. The prepared nanocomposites have been characterized using X-ray Diffraction (XRD), Scanning Electron Microscopy (SEM), UV-VIS. and Fourier Transformation Infrared Spectroscopy (FTIR). The size of nanocomposites heated at 600° C for 4 hour and 8 hour using Scherrer formula comes out to be 5.4 nm and 8 nm respectively. Infrared spectroscopy is also used to determine presence of various functional groups. SEM revealed the polycrystalline structure of the nanocomposite.

Key words: Nanocomposites, Absorption, XRD, SEM, FTIR, UV-VIS.

INTRODUCTION

Aluminium oxide (Al_2O_3) is one of the most versatile ceramic oxides and has been used in a wide range of applications as in electrical, engineering and biomedical areas, depending on its purity and crystallinity [1-3]. Aluminum oxide is commercially produced from bauxite at low cost, but the purity and particle morphology are not suitable for many applications. Preparation of high purity fine alumina particles is of greater importance for advanced applications. Alumina (AlO) nanoparticles have wide range of applications in industrial as well as personal care products. Among other compound semiconductors, rare earth oxide such as Cerium oxide has been applied widely in many fields. Cerium oxide is a semiconductor with a band gap of around 3.0-3.2 eV. Cerium oxide is also well known for its optical properties and can be used to filter ultraviolet rays. Cerium oxide has been widely investigated because of its multiple applications, such as a catalyst, an electrolyte material of solid oxide fuel cells, a material of high refractive index, chemical and mechanical polishing and an insulating layer on silicon substrates [4-6].

The study of composite material has been of great interest from both fundamental and practical point of view. The physical properties like mechanical strength, elasticity, hardness etc. of such materials can be combined to produce material of desired response. Composite have excellent properties such as high hardness, high melting point, high thermal conductivity, good chemical stability and have good potential for various industrial fields [7-10].

MATERIALS AND METHODS

In this investigation, Sol-Gel method is used to prepare AlO-CeO nanocomposites because by this method multi component compounds may be prepared with a controlled stoichiometry by mixing sols of different compounds. It results in small particles, which are easily sinterable. The idea behind sol-gel synthesis is to "dissolve" the

compound in a liquid in order to bring it back as a solid in a controlled manner. The sol-gel method prevents the problems of inhomogeneity which may be occurring with co-precipitation method. In this work mixed ethanol-water solvent (50: 50) was used to dissolve 2 gm aluminium nitrate (AR) and 2 gm cerium nitrate (AR) in the presence of PVA. The mixture was heated at 80° C to form a homogeneous gel solution. The obtained sol was slowly heated to evaporate the solvent and it form a hard homogeneous gel. The pyrolysis process of the final gel was performed at a temperature of 600° C for 4 hour and 8 hour respectively. During the pyrolysis process, aluminium nitrate and cerium nitrate salt simultaneously calcinated and PVA form polymeric network through the outer surface and thus converted them into AlO-CeO nanocomposites. Characterizations of these nanocomposites are done by XRD, TEM and UV-VIS. and FTIR spectrometer.

RESULTS AND DISCUSSION

3.1 XRD Analysis

X-ray diffraction is a non-destructive and analytical method for identification and quantitative analysis of various crystalline forms of prepared nanocomposites, also known as phases of the compound present in the samples. The XRD pattern of AlO-CeO nanocomposites is shown in fig.1(a-b) which were recorded by using PANalytical X'Pert-Pro powder diffractometer employing Cu-K_α radiations in the 2θ range 10°-80°. The particle size of as prepared samples is evaluated by using Scherrer formula [11-13].

$$d = 0.89\lambda/\beta\text{Cos}\theta \quad \text{---------- (1)}$$

where d is average particle size, ß is full width half maxima (FWHM), θ is Bragg's angle and λ is the wavelength of Cu K_α radiations. It was observed that particle size increases on increasing the time of calcination. The particle size comes out to be 5.4 nm and 8 nm respectively for calcination at 4 hour and 8 hour.

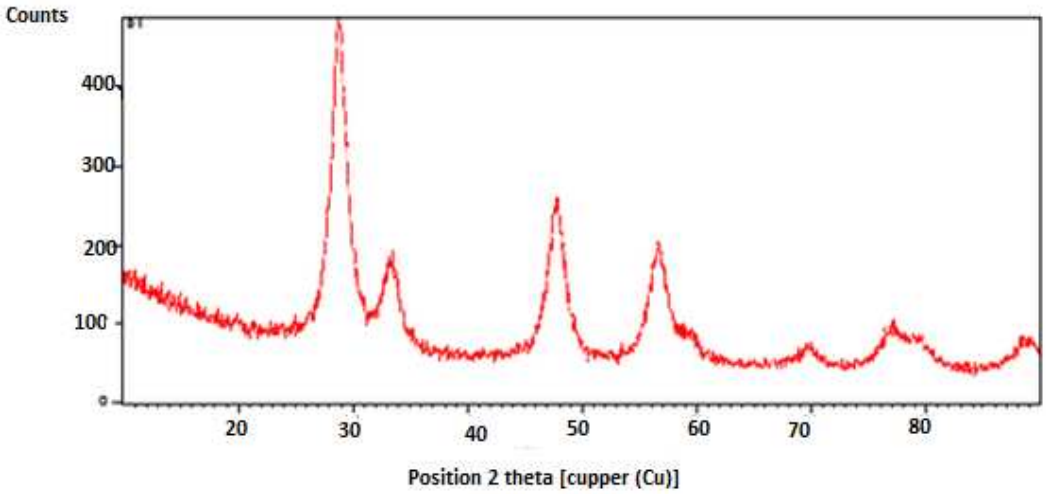

Fig.1(a) XRD pattern of AlO-CeO nanocomposites for 4 hour

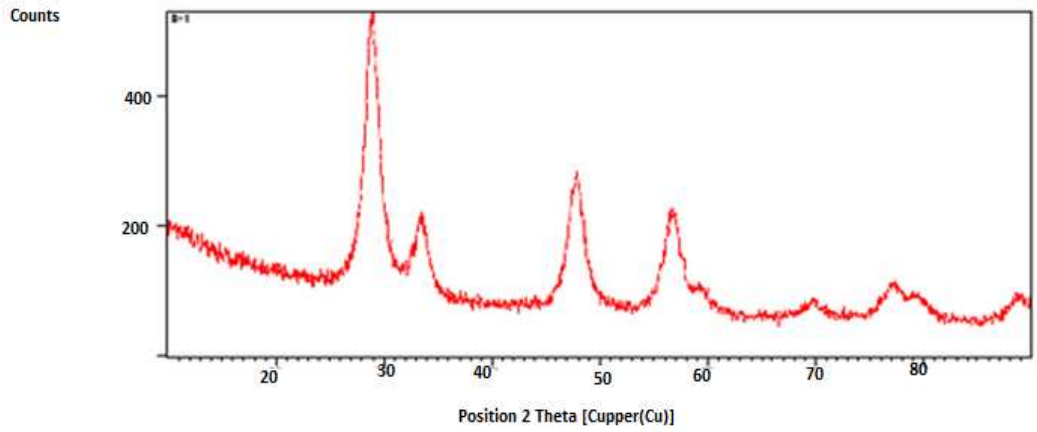

Fig.1(b) XRD pattern of AlO-CeO nanocomposites for 8 hour

3.2 SEM ANALYSIS:

The SEM is used to produce high- resolution imaginings of shapes of substances and to confirm spatial variations in chemical compositions [14-17]. Fig.2(a-b) show SEM images of AlO-CeO nanocomposites calcinated for 4 hour and 8 hour respectively. The images show a general view of the morphology of as prepared nanocomposites.

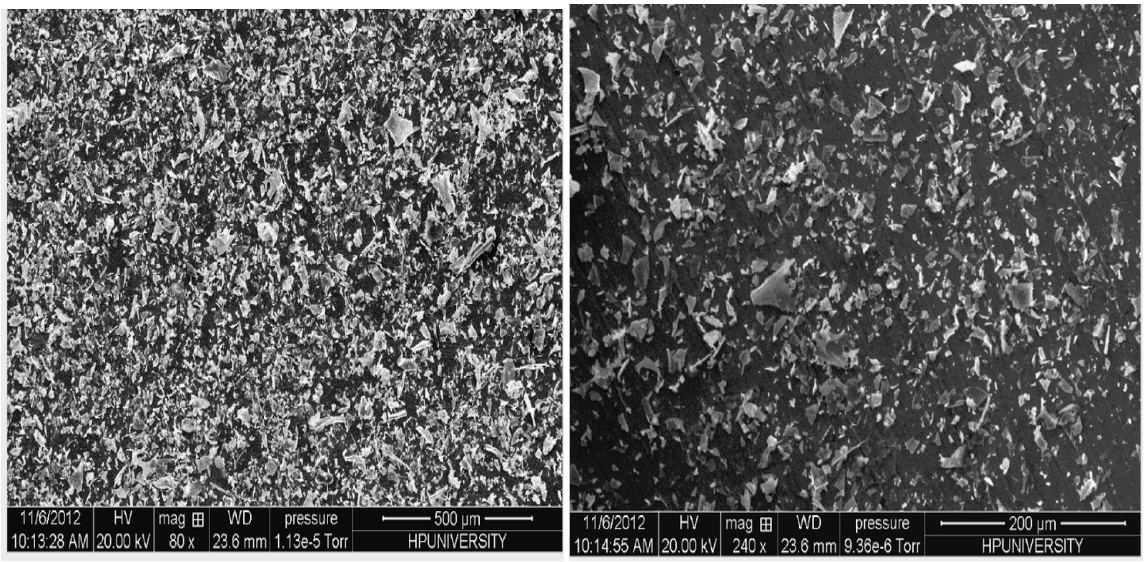

Fig. 2(a) **Fig. 2(b)**

Fig.2(a-b) SEM images of AlO-CeO nanocomposites calcined at 4 h and 8 h

The polycrystalline structure is revealed from the Scanning electron microscope.

3.3 UV-VIS Spectroscopy Analysis:

To evaluate band gap of synthesized nanocomposites UV-VIS spectroscopy is done in the wavelength range 200 nm to 800 nm. The spectra of synthesized nanomaterial calcined for 4 hour and 8 hour at 600° C are shown in fig.3(a-b). At 4 hour, there is absorption peak near to 380 nm but on increasing the time of calcination the peak shift to 330 nm in UV region. Thus there is blue shift in absorption spectra. This confirms the formation of nanocomposites [18-19].

UV spectra provides information about optical band gap of the material. The energy band of the material is related to the absorption coefficient α by the Tauc relation

$$\alpha h\nu = A(h\nu - Eg)^n \qquad \text{----------(2)}$$

where A is constant, hν is photon energy. Eg is band gap and n =1/2 for allowed direct transition [20-21]. The average band gap was estimated from the intercept of linear portion of the $(\alpha h\nu)^2$ vs. hν plots on hν axis.

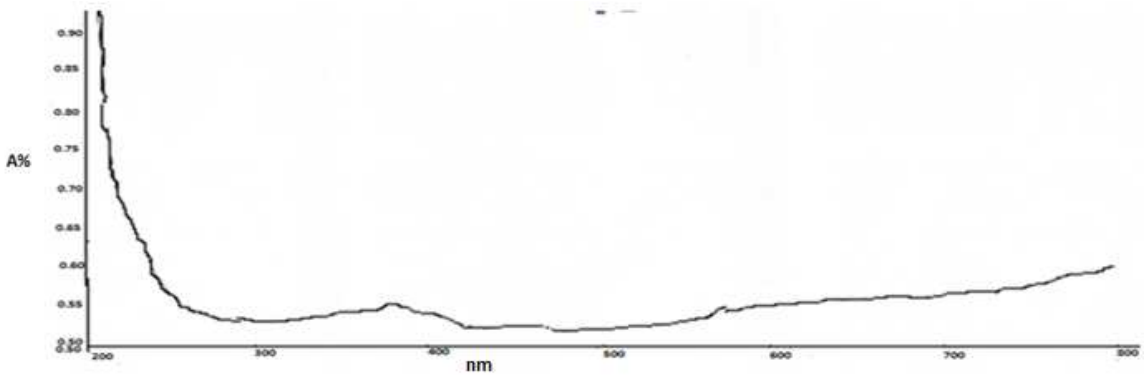

Fig.3(a)

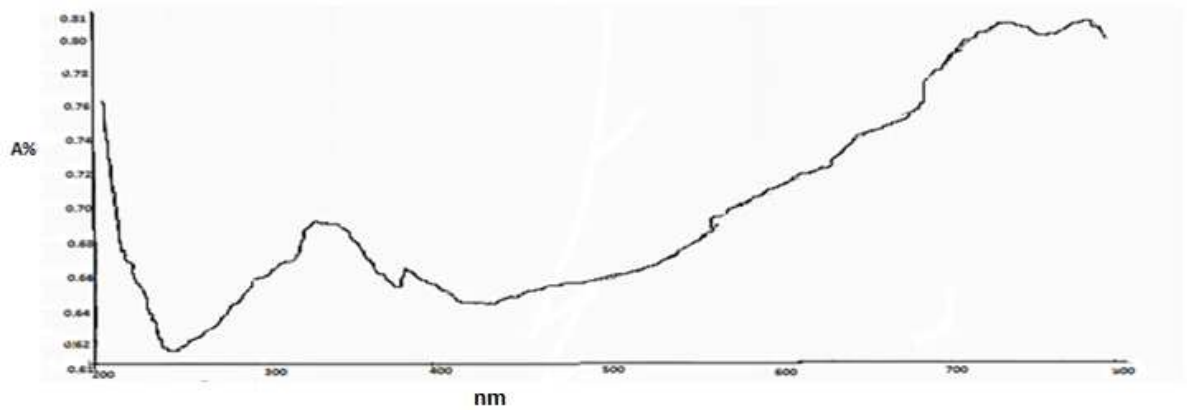

Fig.3(b)

Fig.3(a-b) UV-VIS images of AlO-CeO nanocomposites calcined at 4 h and 8 h respectively.

3.4 FTIR Spectroscopy:

Infrared spectroscopy is used to determine presence of particular functional group [22]. The infrared spectroscopic study of the nanocomposites were done using Perkin Elmer- spectrum FTIR Spectrometer in the wave number range 400-4000 cm^{-1}. FTIR spectra of AlO-CeO nanocomposites are shown in fig.4(a-b). FTIR Spectra calcined for 4 hour at 600° C shows peaks at 3401 cm^{-1}, 2339 cm^{-1}, 1574 cm^{-1}, 1414 cm^{-1}, 1019 cm^{-1}, 864 cm^{-1}, 485 cm^{-1} as shown below.

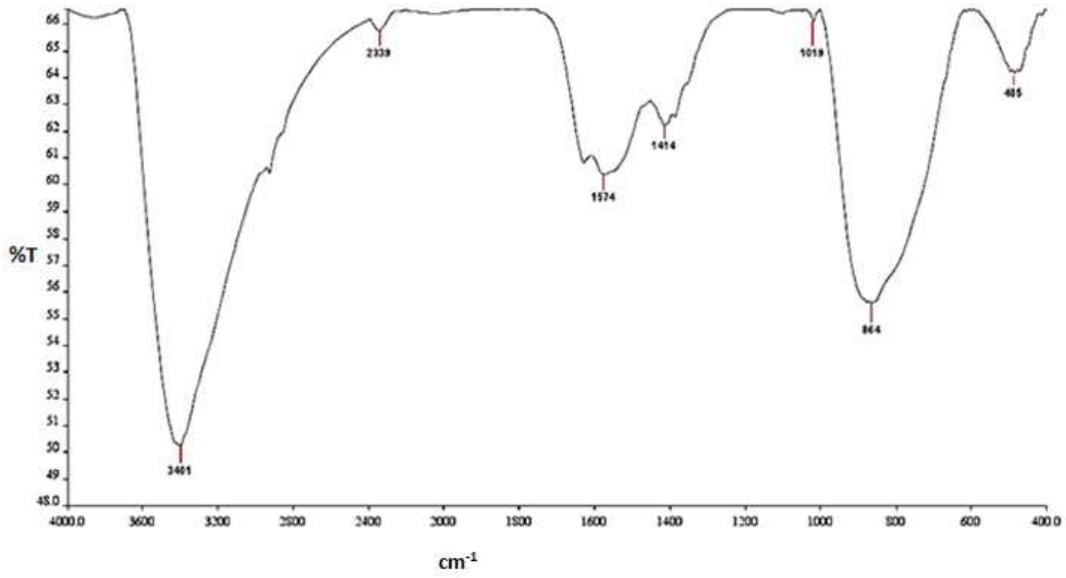

Fig.4(a) FTIR image of AlO-CeO nanocomposites calcined at 4 hour

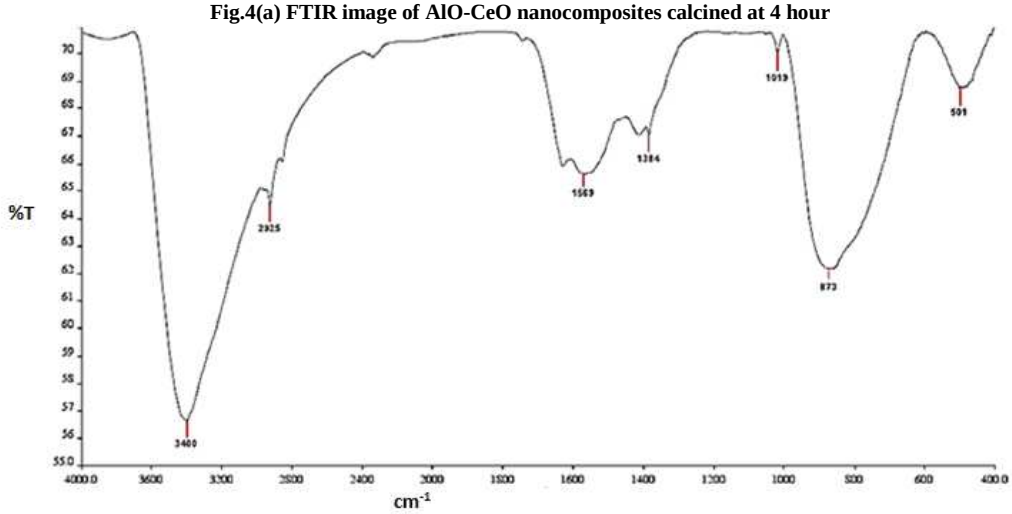

Fig.4(b) FTIR image of AlO-CeO nanocomposites calcined at 8 hour

A broad band at 3401 cm^{-1} corresponds to stretching mode of OH group which is contributed by water contents. The peak at around 2339 cm^{-1} is due to C=C band. Band around 1574 cm^{-1} is due to deformation vibration of H_2O molecule. Band around 1100 cm^{-1} may be due to single C-C bond stretching mode [23-25].

FTIR Spectra of prepared nanomaterial calcined for 8 hour at 600° C shows peaks at 3400 cm^{-1}, 2925 cm^{-1}, 1569 cm^{-1}, 1384 cm^{-1}, 1019 cm^{-1}, 873 cm^{-1}, 501 cm^{-1} respectively. The peak at 2925 cm^{-1} is due to C-H band. The peak around 1384 cm^{-1} is due to C-O band. Band around 870 cm^{-1} may be due to Ce-O stretching vibration and band around 500 cm^{-1} is due to Al-O stretching vibration.

CONCLUSION

AlO-CeO nanocomposites have been prepared successfully by Sol-Gel technique. The crystalline size of synthesized nanocomposites which are calcined for 4 hour and 8 hour at 600° C were evaluated by using Scherrer formula and its comes out to be 5.4 nm and 8 nm respectively. SEM images show a general view of the morphology of as prepared nanocomposites. It is shown that synthesized nanocomposites are polycrystalline in nature. Also FTIR Spectra confirms the presence of C=C and C-O, C-C bond stretching mode.

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