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

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ABSTRACT

In the present investigation, main emphasis has been given to synthesize AlO-ZnO 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, zinc 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 36.4 nm and 15.20 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

Nanoparticles are classified as being materials in which at least one dimension of the material is less than 100 nanometers in diameter. Nanoparticles are becoming an area of research interest due to their unique properties, such as having increased electrical conductivity, ductility, toughness, and formability of ceramics, increasing the hardness and strength of metals and alloys, and by increasing the luminescent efficiency of semiconductors. Nanoparticles are heavily used in an industrial application because they can be used to manufacture lightweight, strong materials as well as acting as pigments in products such as paints, sunscreens, and cosmetics. Because nanoparticles have a large surface area to volume ratio, the use of nanoparticles in both industry and daily life is greatly increasing in realms that include advancing the quality of everyday materials and processes, improving the function of electronics and information technology, allowing more sustainable energy applications, and acting as key players in environmental remediation applications [1-3]. 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. 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. Aluminum oxide is also very wear-resistant, has good thermal conductivity, resists strong acid and alkali containing materials, is easily shaped, and has high strength and stiffness which makes it a prime material to use in making products that include high temperature electrical insulators, high voltage insulators,

thermometry sensors, wear pads, ballistic armor, and grinding media Among other compound semiconductors, Zinc oxide has been applied widely in many fields [4-6]. Zinc oxide is also well known for its optical properties and can be used to filter ultraviolet rays. Zinc 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, gas sensors, Thin film applications, Textiles, Optical coating and antibacterial [7-9].

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 [10-12].

MATERIALS AND METHODS

In this investigation, Sol-Gel method is used to prepare AlO- ZnO 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 [13]. 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 zinc 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 zinc nitrate salt simultaneously calcinated and PVA form polymeric network through the outer surface and thus converted them into AlO-ZnO nanocomposites. Characterizations of these nanocomposites are done by XRD, TEM and UV-VIS. and FTIR spectrometer.

RESULTS AND DISCUSSION

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-ZnO 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 [14-20].

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

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 36.4 nm and 15.20 nm respectively for calcination at 4 hour and 8 hour.

SEM ANALYSIS:

The SEM is used to produce high- resolution imaginings of shapes of substances and to confirm spatial variations in chemical compositions. Fig.2(a-b) show SEM images of AlO- ZnO nanocomposites calcinated for 4 hour and 8 hour respectively. The images show a general view of the morphology of as prepared nanocomposites. The morphology of both nanocomposites reveals that they form nanoclusters on annealing at 4h and 8h [21-23].

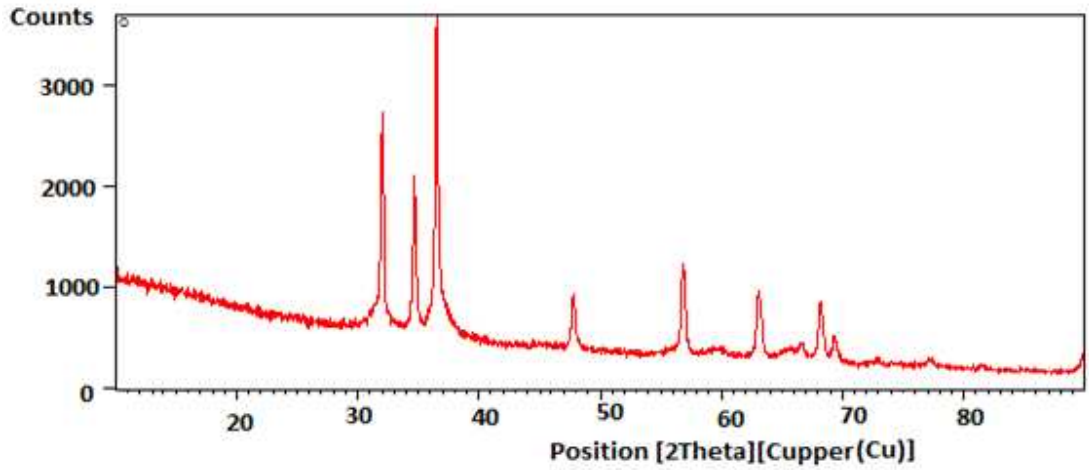

fig.1(a) XRD pattern of AlO-ZnO nanocomposites for 4 hour

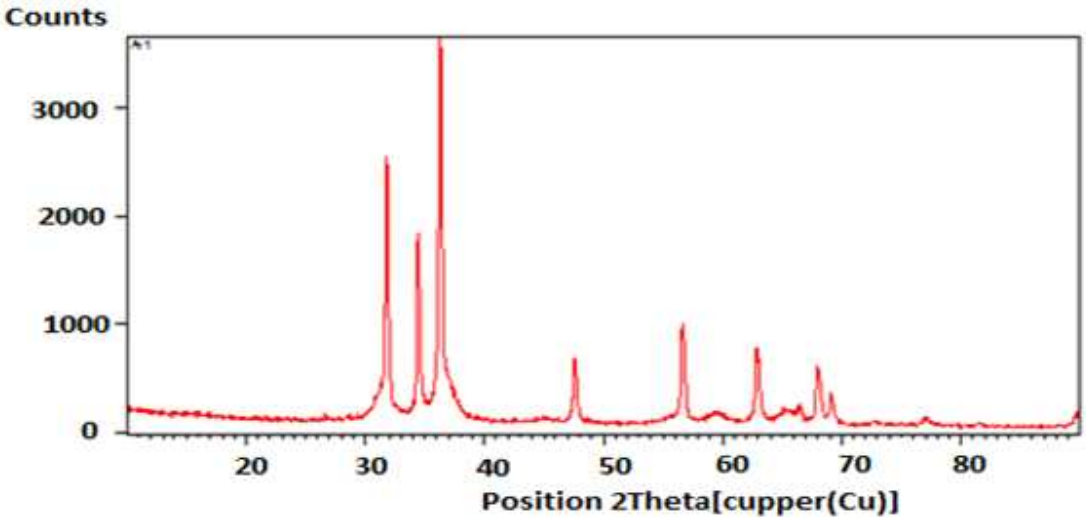

Fig.1(b) XRD pattern of AlO-ZnO nanocomposites for 8 hour

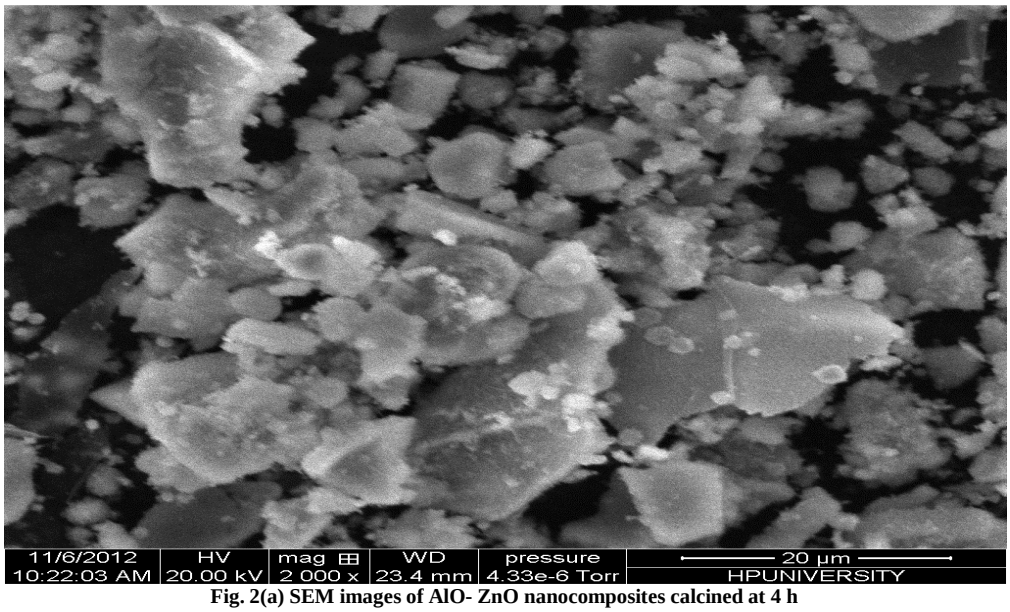

Fig. 2(a) SEM images of AlO- ZnO nanocomposites calcined at 4 h

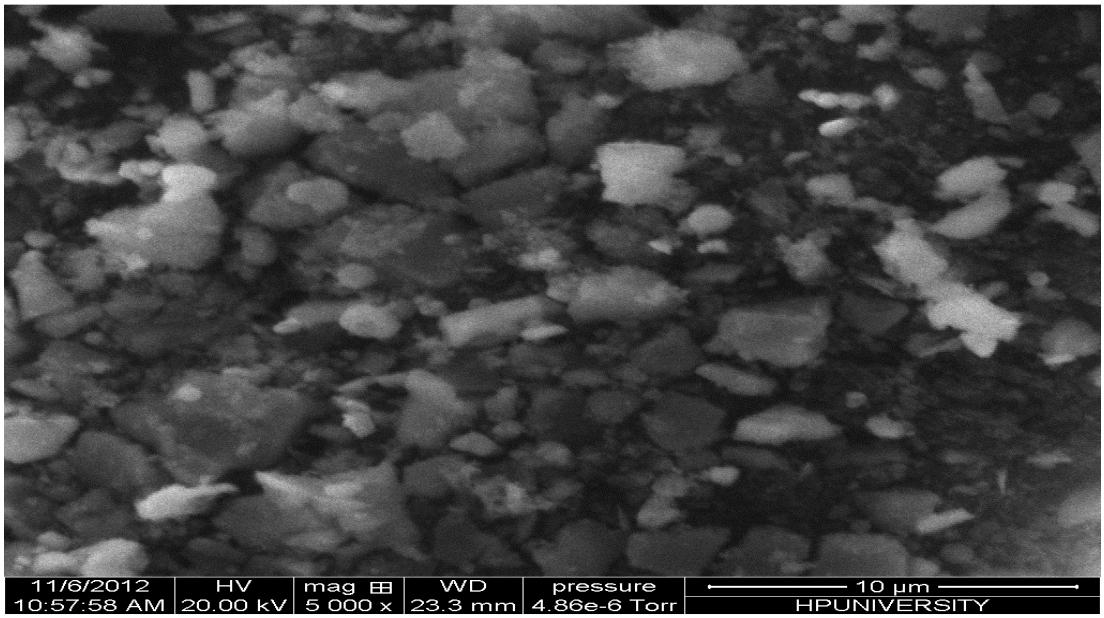

Fig. 2(b) SEM images of AlO- ZnO nanocomposites calcined at 8 h

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 240 nm and 380 nm. The absorption first decreases fast in ultraviolet region and then in the visible region it increases linearly with small slope. On increasing the time of calcinations, the absorption peaks are at 230 nm, 280 nm, 380 nm in UV region and at 560 nm in visible region. The study of UV-VIS radiation absorption is an important tool for the evaluation of the changes in the produced semiconductor material by different treatments. The band gap energy was calculated on the basis of maximum absorption band of Alo-CeO nanoparticles according to equation

$E_g=1240/\lambda$ ----------(2)

Where E_g is the band gap energy and λ is the lower cutoff wavelength (nm) of the nanocomposite. The value of the band gap when calcinated at 600^{O}c at 4h and 8h comes out to be 4.2 eV and 4.4 eV using this equation (2).

Since there is blue shift in absorption spectra when we calcinate the samples from 4h to 8h , this confirms the formation of nanocomposites [24-27].

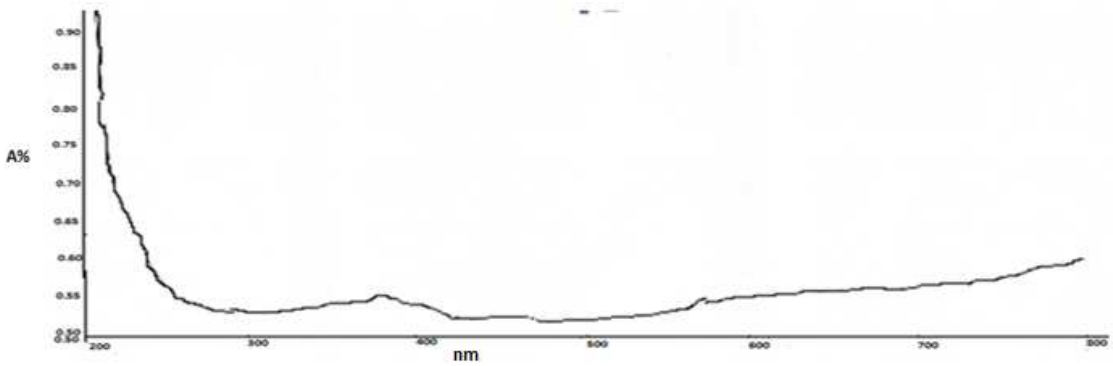

Fig.3(a) UV-VIS image of AlO-ZnO nanocomposites calcined at 4 h

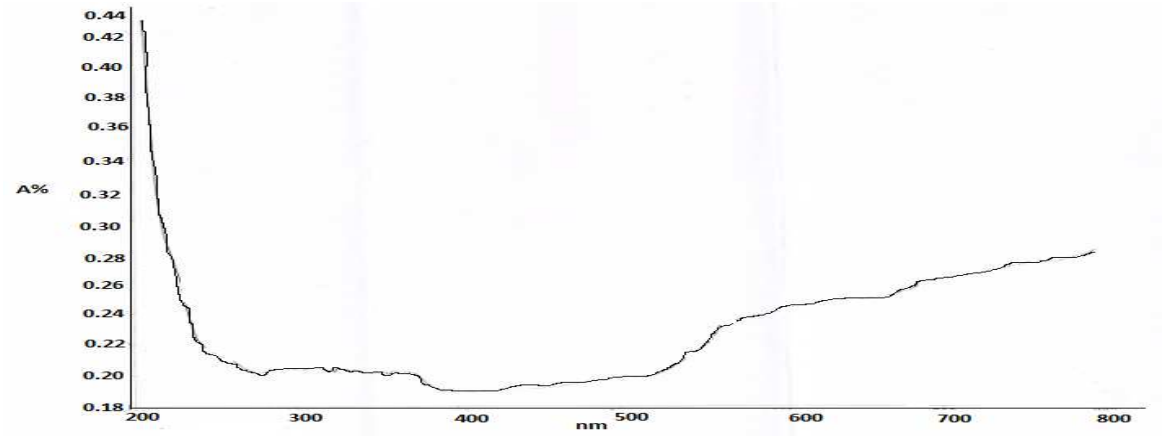

Fig.3(b) UV-VIS image of AlO-ZnO nanocomposites calcined at 8 h

A UV spectrum 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

$$\alpha h\nu = A\,(h\nu - Eg)^{n} \qquad \text{----------(3)}$$

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

FTIR Spectroscopy:

Infrared spectroscopy is used to determine presence of particular functional group. 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-ZnO nanocomposites are shown in fig. 4(a-b). FTIR Spectra calcined for 4 hour at 600° C shows peaks at 3432 cm^{-1}, 2927 cm^{-1}, 2341 cm^{-1}, 2078 cm^{-1}, 1631 cm^{-1}, 1570 cm^{-1} 1410 cm^{-1} 1093 cm^{-1} 1020 cm^{-1} 963 cm^{-1} 690 cm^{-1} 561 cm^{-1} 506 cm^{-1} 467 cm^{-1} 440 cm^{-1} as shown below. FTIR Spectra of prepared nanomaterial calcinated for 8 hour at 600° C shows peaks at 3434 cm^{-1}, 2340 cm^{-1}, 2089 cm^{-1}, 1635 cm^{-1}, 1563 cm^{-1}, 1411 cm^{-1}, 1101 cm^{-1} 1021 cm^{-1} 756 cm^{-1} 690 cm^{-1} 555 cm^{-1} 500 cm^{-1} 449 cm^{-1} respectively.

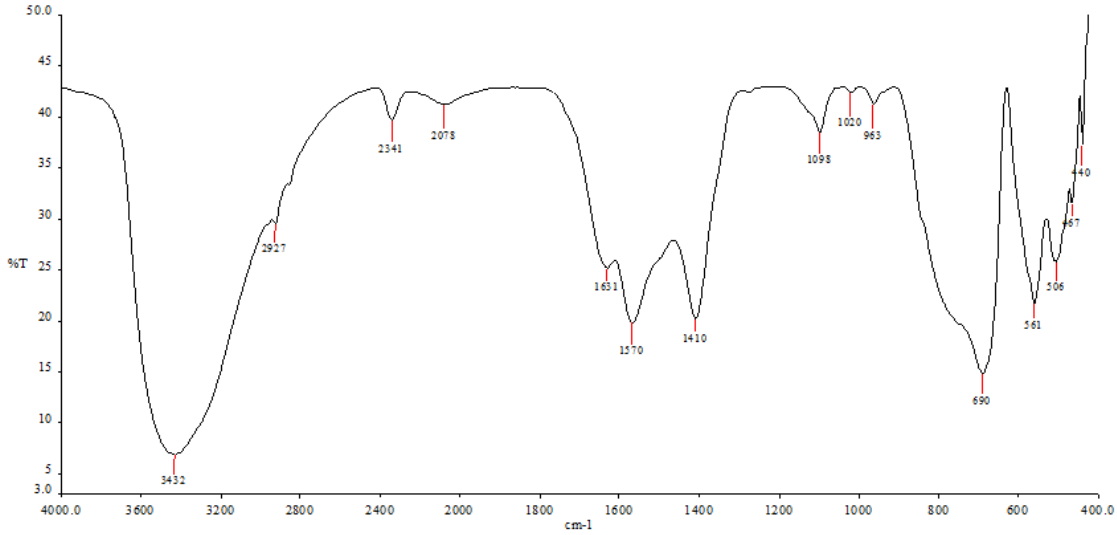

Fig.4(a) FTIR image of AlO-ZnO nanocomposites calcined at 4 hour.

A broad band at 3401 cm^{-1} corresponds to stretching mode of OH group which is contributed by water contents. The peak around 2927 cm^{-1} may be due to C-H bond .The peak at around 2339 cm^{-1} is due to C=C band. Band around 1631 cm^{-1} is due to O-H bending of absorbed water. Band around 1574 cm^{-1} is due to deformation vibration of H_2O

molecule. Band around 1410 cm^{-1} may corresponds to asymmetric stretching of C=O bonds. Band around 1100 cm^{-1} may be due to single C-C bond stretching mode. Band at 690 cm^{-1} may corresponds to stretching vibration of M-O-M where M corresponds to metal occupying tetrahedral and octahedral. The absorption band at wave number around 561 cm^{-1} represents the Al-O stretching mode. The absorption band around 457 cm^{-1} corresponds to stretching mode of Zn-O. [28-31].

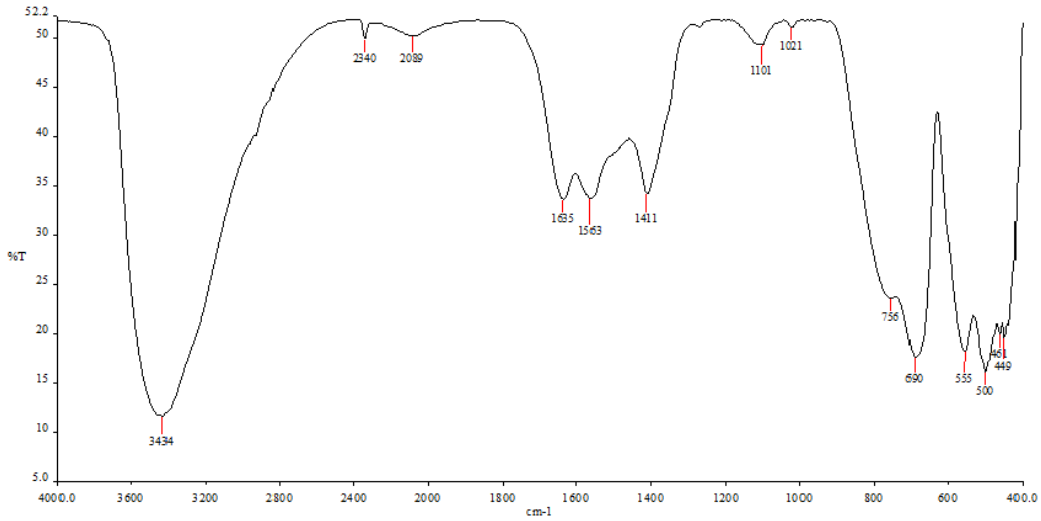

Fig.4(b) FTIR image of AlO-CeO nanocomposites calcined at 8 hour.

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 Zn-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 36.4 nm and 15.20 nm respectively. Here particle size decreases when we increase the time of calcination from 4h to 8h. SEM images show a general view of the morphology of nanocomposites when it is calcinated for 600^{o}C at 4h and 8h which shows that synthesized nanocomposites are polycrystalline in nature. Band gap of as prepared nanomaterials is 4.2 eV and 4.4 eV respectively. Also FTIR Spectra confirms the presence of C=C and C-O, C-C bond stretching mode.

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