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Effect of calcination on optical properties and morphology of NiO-CuO NanoComposites

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ABSTRACT

A novel NiO-CuO nanocomposite has been synthesized by a sol-gel method based on polymeric network of polyvinyl alcohol (PVA). In this work mixed solvent of 50:50 ethanol water was used to dissolve cupric nitrate, nickel nitrate and PVA. The mixture was heated to 80° C to form a homogeneous gel solution. The obtained gel was slowly heated to evaporate the solvent to form a hard homogeneous gel. The hard gel was calcinated at temperature 600° C for 4 hours and 6 hours and converted into nanocomposite. The prepared nanocomposites have been characterized using X-Ray Diffraction (XRD), FTIR, UV-VIS. In the observed spectral features, the peak position, intensity and bandwidth were related to some structural properties of investigated samples. The size of NiO-CuO nanocomposites increases with increase in temperature. Infrared spectroscopy is also used to determine presence of various functional groups. XRD revealed the polycrystalline structure of the nanocomposite. SEM gives the morphology of the nanocomposites.

Keywords: NanoComposite, Synthesis, XRD, FTIR, UV-VIS.

INTRODUCTION

There are various methods for the preparation of NanoComposites but Sol–Gel method has been largely used for the preparation of inorganic oxide NanoComposites. The Sol-Gel technique is low temperature route that provide good homogeneity for the preparation of metal oxide NanoComposites. 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.

NiO is the most exhaustively investigated transition metal oxide. It has a density 6.67 gm/cm³, green crystalline solid having melting point 1955^oC. It is NaCl-type antiferromagnetic oxide semiconductor. It has many applications such as solar thermal absorber, as catalyst, photoelectrolysis and electrochromic device. Nickel oxide is also a well-studied material as the positive electrode in batteries. NiO is a p-type semiconductor and its electrical conduction is almost entirely contributed from electron hole conduction. NiO is preferred for high electrochromic efficiency, low cost and high dynamic dispersion [1-3]. Among another oxide nanomaterials, Copper oxide

nanoparticles are used in a wide range of applications such as gas sensors, magnetic storage media, batteries, solar energy transformation, semiconductors, heterogeneous catalysis and field emission[4-6]. Copper oxide is of great interest in semiconductor physics. Copper forms two well known stable oxides, cupric oxide (CuO) and Cuprous oxide (Cu₂O). These two oxides have different physical properties, different colors, crystal structures and electrical properties. CuO is used for sensing applications. CuO has many unique features like high specific surface area, chemical stability, electrochemical activity, high electron communication features. Cupric oxide is a p-type semiconductor with an in direct band gap of 1.21-1.51 eV. CuO has been used as a basic material in High Tc superconductors as the superconductivity in these classes is associated with CuO bondings. The possibility of low cost production methods and the good electrochemical properties make CuO one of the best materials for electrical, optical, sensing etc. and also used as CO oxidation of automobile exhaust gases. Cuprous Oxide (Cu₂O) is p-type direct band gap, 11-V1 semiconductor with a band gap of nearly 2eV [7-9].

The study of composite material is a mixture consisting of at least two phases of different chemical composition. It has been of great interest from both fundamental and practical point of view. The physical properties of such materials can be combined to produce material of desired response. Optical or magnetic characteristics can change upon the decrease of particle sizes to very small dimensions, which are in general of major interest in the area of nanocomposite materials. Composites have excellent properties such as high hardness, high melting point, low density, low coefficient of thermal expansion, high thermal conductivity, good chemical stability and improved mechanical properties such as higher specific strength, better wear resistance and specific modulus and have good potential for various industrial fields the synthesized nanoparticles were characterized by XRD, TEM and UV-VIS, FTIR Spectrometer

MATERIALS AND METHODS

In this investigation, Sol-Gel method is used to prepare NiO-CuO Nanocomposites because by this method, multicomponent 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 in homogeneity which may be occurring with co-precipitation method [10-12].

In this work mixed ethanol-water solvent (50:50) was used to dissolve 4 gm cupric nitrate, 4gm nickel nitrate and 8 gm PVA, the mixture was heated to 80°C to form a homogeneous sol solution. The obtained sol was slowly heated to evaporate the solvent and it form a hard homogeneous gel. The Pyrrolysis of the final gel was performed at a temperature of 600°C for 4 hours and 6 hours. During the Pyrrolysis process the PVA polymeric network through the outer surface, nickel and cupric nitrate salts simultaneously calcinated and converted into NiO-CuO nanocomposite. The obtained samples were crushed to prepare a fine powder. Characterizations of these nanocomposites are done by XRD, TEM and UV-VIS. and FTIR spectrometer.

RESULTS AND DISCUSSION

1.1 XRD STUDIES:-

The XRD pattern of NiO-CuO NanoComposites which were calcinated at 600^{0} C for 4 hours and 6 hours are shown in Fig. 1 (a) and 1 (b).Both the samples which are calcinated for 4 h and 6 h are polycrystalline in nature. The particle size of the samples was found using Scherrer formula

$d=0.89\lambda/\beta Cos\theta$

Where d= average particle size. β is full width at half maxima (FWHM), θ is Bragg's angle and λ is the wavelength of Cu K_a radiations. The particle size when calcinated at 600^o C for 4 h is 17.1 nm whereas when calcinated at 600^o C for 6 h comes out to be 21.2 nm. It is seen from XRD that particle size increases when we increase the time of calcinations [13-15]. In fig 1.(a) a series of characteristic peaks (104), (119) ,(101), (111) ,(202), (110), (202), (113), (103) and in fig.1.(b) a series of characteristic peaks are (112), (214), (101), (110) and (113) are noticed.



Fig.1 (b) XRD pattern of NiO CuO Nanocomposites calcinated at 600 ⁰ C for 6 hour

50 Position [2Theta] (Copper (Cu))

40

1.2 UV-VIS SPECTRAL STUDIES:-

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0



Fig.2 (a) UV-VIS image of NiO CuO NanoComposites calcinated at 600 ⁰ C for 4 h



The UV-VIS study of Nanocomposites NiO -CuO were done using Hitachi 330 spectrophotometer in the wavelength range 200 to 800 nm. UV-VIS spectra of NiO CuO nanocomposites are shown in fig 5 and fig 6. In NiO- CuO nanocomposites when heated at 600°C for 4 h, absorption first decreases up to 260 nm in ultraviolet region then it increases sharply and then there is a peak at 390 nm and again rises linearly. In 6 h spectra, there is a fast decrease in absorption up to 260 nm in ultraviolet region, then it increases sharply. There are two peaks at 340 nm in ultraviolet region and at 600 nm and 650 nm in visible region and then rises linearly. UV-VIS spectra show that the absorption of NiO- CuO nanocomposites when calcinated at 60 not increases as sharp as when calcinated for 4 h [16-19]. Cut off wavelength in both the cases is same is 280 nm. The bandgap of the nano composite in both cases is calculated on the basis of the formulae as given below:

Eg= $1240/\lambda$ (eV)

which comes out to be 4.4 eV. Thus there is no change in bandgap when we calcinated the nanocomposites from 4h to 6h.

1.3 FTIR SPECTRA ANALYSIS :-



Fig.3 (a) FTIR image of NiO CuO NanoComposites calcinated at 600 ⁰ C at 4 hour



Fig.3 (b) FTIR image of NiO- CuO nanocomposites calcinated at 600 ° C at 6 hour

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 NIO-CuO NanoComposites are shown in fig. 4(a-b). FTIR Spectra calcinated for 4 hour at 600° C shows peaks at 3400 cm⁻¹, 2900 cm⁻¹, 2150 cm⁻¹, 1456 cm⁻¹, 1354 cm⁻¹, 582 cm⁻¹, 534 cm⁻¹ as shown above. FTIR Spectra of prepared nonomaterial calcinated for 6 hour at 600° C shows peaks at 3400 cm⁻¹, 1728 cm⁻¹, 1456 cm⁻¹, 1020 cm⁻¹, 840 cm⁻¹, 701 cm⁻¹, 667 cm⁻¹ and 533 cm⁻¹ respectively.

A broad band at 3400 cm⁻¹ corresponds to stretching mode of OH group which is contributed by water contents. The peak around 2900 cm⁻¹ may be due to C-H stretching bond. The peak around 2150 cm⁻¹ may be due to C=C bond. The peak around 1456 cm⁻¹ may be due to C=C bond. The peak around 1354 cm⁻¹ may be due to C-O bond stretching mode. The peak around 582 cm^{-1} and 534 cm^{-1} may be due to Cu-O stretching viberational mode.

A broad band at 3400 cm⁻¹ corresponds to stretching mode of OH group which is contributed by water contents. The peak around 1728 cm⁻¹ may be due to OH group in the metal alkoxides present in the gel and 1456 cm⁻¹ may be due to C=C bond. Bands around 1020 cm⁻¹

May be due to single C-O bond stretching mode. Band around 701 cm⁻¹ may be due to Metal Oxygen band. Bands at 619 may be due to stretching viberation of M-O-M metal oxide bond where M corresponds to metal occupying tetrahedral and octahedral. Bands around 840 may be due to Ce O stretching viberation [20-23].

1.4SEM:-

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 NIO- CuO 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 particles are spherical , regular and uniform in shape. They reveal randomly oriented aggregates and formation of nano crystalline material. The size of nano aggregates is more when we increase the time of calcinations from 4h to 6h at 600^{0} C [24-25].



Fig. 4(a) SEM images of NIO- CuO NanoComposites calcinated at 600 ° C at 4 h



Fig. 4(b) SEM images of NIO-CuO NanoComposites calcinated at 600 ° C at 6 h

CONCLUSION

NiO- CuO NanoComposites have been prepared successfully by Sol-Gel technique. The crystalline size of synthesized nanocomposites which are calcined for 4 hour and 6 hour at 600° C were evaluated by using Scherrer formula and its comes out to be 17.1 nm and 21.2 nm respectively. Here particle size increases when we increase the time of calcination from 4h to 6h. SEM images show a general view of the morphology of NanoComposites. When it is calcinated for 600°C at 4h and 6h, it shows that the synthesized NanoComposites are polycrystalline in nature. Also FTIR Spectra confirms the presence of C=C and C-O, C-C bond stretching mode. From UV-VIS spectra it is seen that there is no change in band gap (4.4 eV) when we calcinate it from 4 h to 6 h.

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