Synthesis and Characterization of ZnO Nanoparticles

T.Enkhtuya[1],* G.Tsermaa[1], G.Batdemberel[1], P.Munkhbaatar[1], Sh.Chadraabal[1], N.Javkhlantugs[2]

[1] *School of Applied Science, Mongolian University of Science and Technology, Baga toiruu 34, Sukhbaatar district,Ulaanbaatar, 14191, Mongolia,*

[2] *Laboratory of Nanotechnology, School of Engineering and Applied Sciences, National University of Mongolia. University Street 1, Sukhbaatar district,Ulaanbaatar, 14201, Mongolia.*

This study is focused on hydrothermal synthesis method of zinc oxide nanoparticles by using ammonia and zinc chloride. Synthesized nanoparticles have been characterized by using X-ray Diffraction, UV-vis and Photon Cross Correlation (PCCS) spectroscopies, TEM analysis. ZnO thin films were prepared by spin coating method from the solution of ZnO nanoparticles. XRD peak corresponds to hexagonal structure with cell parameter a=0.326 nm and c=0.522 nm. The grain size of zinc oxide nanoparticles has been determined 43.3 nm using the Scherrer formula. As a result of PCCS measurement, synthesized zinc oxide nanoparticles have the mean diameter of ~96 nm of main particles with particle size distribution range of from ~90 nm to ~99 nm. As the result of TEM, the respective size of nanoparticles was around ~96 nm. ZnO nanoparticles were coated on glass and indium-tin oxide (ITO) substrates. The photocatalytic activities were studied ITO substrates with ZnO thin film. ZnO thin films were used as catalyst for reaction Cr(VI) into Cr(III).

Keywords: zinc oxide, hydrothermal method, particle size, photocatalysis.

INTRODUCTION

Zinc oxide (ZnO) is one of the multifunctional inorganic has drawn increasing attention in recent years due to its prominent physical and chemical properties, such as chemical stability, low dielectric constant, high luminousis transmittance, high catalysis activity, effective antibacterial and bactericide, intensive ultraviolet and infrared absorption [1]. The optoelectronic properties of ZnO nanoparticles depend on particle sizes, which can be controlled by reaction conditions during their preparation and by attachment of organic compounds onto the surfaces. ZnO has many nanostructures among all one-dimensional. ZnO nanocrystals with various shapes included one-dimensional (rod, tube, wire and nail), two-dimensional (sheet, hexagon, tower and comb) and multi-dimensional (flower), have been successfully demonstrated [2].

ZnO nanoparticles can be prepared on a large scale at low cost by simple solution-based synthesis methods, such as chemical precipitation ([3-5]) sol-gel synthesis ([6-7]), and solvothermal/hydrothermal reaction [8]. However, agglomeration and secondary growth often occur in these ZnO nanoparticles when we dry the wet particles separated from the reaction solution. This is because large numbers of hydroxyl groups exist on the wet particle surface. Hydrothermal technique is promising alternative synthetic method because of the low process temperature and very easy to control the particle size. The hydrothermal process have several advantage over other growth processes such as use of simple equipment, catalyst-free growth, low cost, large area uniform production, environmental friendliness and less hazardous. The low reaction temperatures make this method an attractive one for microelectronics and plastic electronics. This method has also been successfully employed to prepare nanoscale ZnO and other luminescent materials. The particle properties such as morphology and size can be controlled via the hydrothermal process by adjusting the reaction temperature, time and concentration of precursors [9].

Zinc oxide crystallizes in two main forms, hexagonal wuartzite and cubic zincblende. The wuartzite structure is most stable at ambient conditions and thus most common. The zincblende form can be stabilized by growing ZnO on substrates with cubic lattice structure. In both cases, the zinc and oxide centers are tetrahedral, the most characteristic geometry for Zn [10]. Under conventional conditions, ZnO has the wuartzite structure, which has a hexagonal unit cell with space group C6mc and lattice parameters a=0.3296, and c=0.52065 nm. The oxygen anions and Zn cations form a tetrahedral unit.

MATERIALS AND METHODS

In order to synthesize the ZnO nanoparticles we used raw materials zinc chloride $ZnCl_2$ (0.1M) and ammonia NH_3 (25%). Samples were calculated using the following equation:

$$ZnCl_2+2NH_3+H_2O=2NH_4Cl+ZnO$$

* Electronic address: enkhtuya@must.edu.mn

Zinc chloride, 1.083 g (0.1 mol), was dissolved in 80 ml distilled water, then the solution was kept under constant stirring using magnetic stirrer for half an hour to completely dissolve the zinc chloride. After complete dissolution of zinc chloride, 4ml of ammonia solution (25%) was added under constant stirring, drop by drop. The reaction was allowed to proceed for 24 hours. After the completion of reaction the solution was poured into the crystallization vessel, then this vessel was heated to 90^0C in an ordinary laboratory oven, and kept in it for specific times. Subsequently, cooled down to the room temperature normally (using magnetic stirrer).

The resultant solution has been thoroughly washed with distilled water more than five times, washing carried out to remove the unwanted particles and to ensure that we got just ZnO nanoparticles. After that it was dried in the 90^0C in laboratory oven, and then a white powder was obtained, then grinded for uniformities of the powder.

X-ray diffraction data for sample prepared was recorded by using (Cu, Kα) radiation of wavelength (1.5419 Å). We used the the X-Ray powder diffractometer.

Samples of ZnO for PCCS analysis and TEM (JEM-2100F) were put into ethanol (99.5%) and prepared suspension.

The reduction of Cr(VI) was carried out by three different catalysts. For the catalysis three samples were prepared. First, ZnO nanoparticle were put into Cr(VI) solution. Also, ZnO thin film on ITO (indium-tin) surface, and ZnO thin film on glass substrates were produced. Absorbance was measured by UV-vis (UV-1650PC).

RESULTS AND DISCUSSION

A. XRD Result

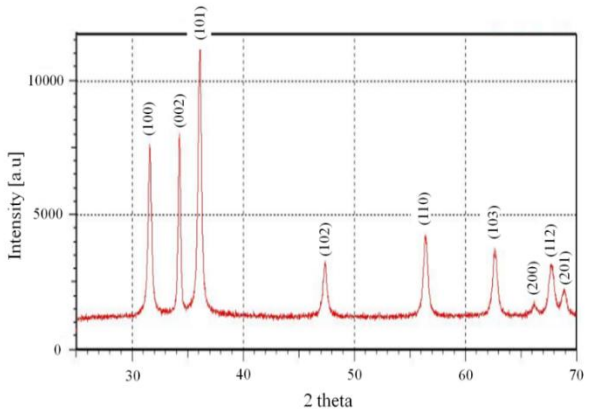

Fig 1. XRD patterns of ZnO nanoparticles.

Figure 1 shows that XRD image of ZnO nanoparticles. X-ray diffraction peaks agreed with the reported JCPDS data (JCPDS Card No. 36-1451) and compared this results with data published in many articles get the sample position of the peaks and all the samples polycrystalline and correspond to hexagonal structure with lattice spacing $a = 0.325nm$ and $c = 0.521nm$ (Laudise, R.A, 1986).

XRD peak corresponds to hexagonal structure with cell parameter a=0.326 nm and c=0.522 nm. The grain size of zinc oxide nanoparticles has been determined 43.3 nm from the full with at half maximum (FWHM) of (101) diffraction peak using the Scherrer formula.

$$D = \frac{0.89\lambda}{\beta \cos\theta}$$

Where λ the wavelength (Cu, Kα), β is the (FWHM), θ is the diffraction angle.

B. TEM Studies of ZnO nanoparticles

TEM images of the synthesized ZnO nanopartiles is shown in Fig 2. As can be seen from Fig.2, ZnO nanoparticles have average particle sizes as ~96 nm.

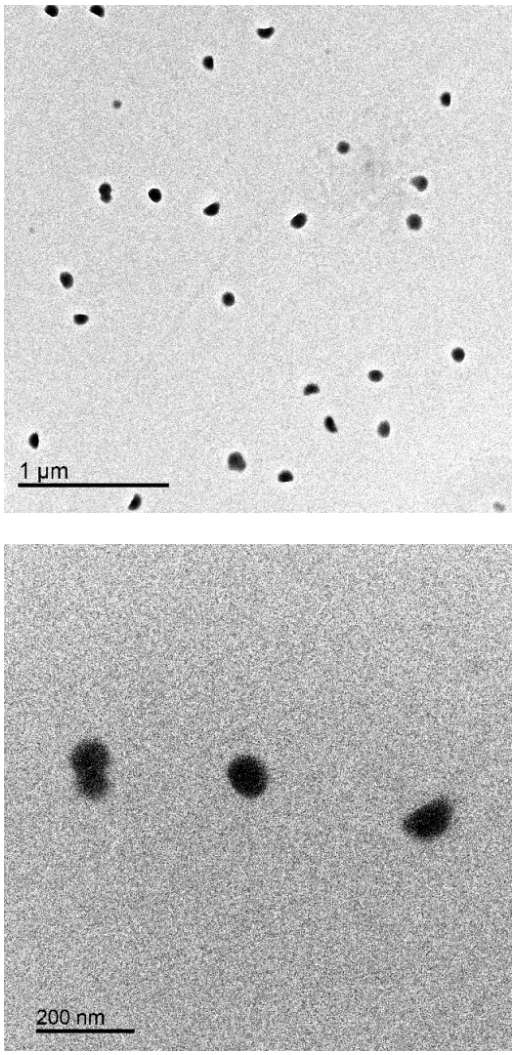

Fig 2. TEM images of ZnO nanoparticles

C. PCCS Analysis

The data of PCCS analysis was calculated by WINDOX 5. Particle size and cumulative distribution of ZnO are depicted in Fig.3

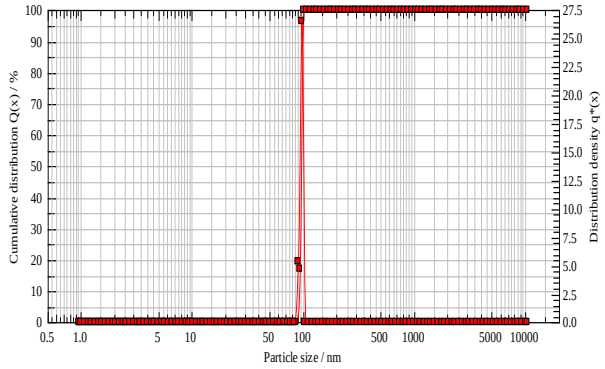

Fig 3. Cumulative and density distribution of ZnO nanoparticles

As a result of PCCS measurement, synthesized zinc oxide nanoparticles have the mean diameter of ~96 nm of main particles with particle size distribution range of from ~90 nm to ~99 nm.

D. UV-vis measurement

The photocatalytic activities of these ITO substrate with ZnO thin film were studied.

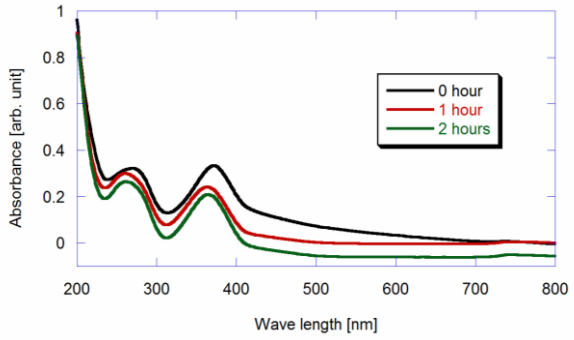

Figure 4. UV spectrum of $K_2Cr_2O_7$ solution in ZnO thin film on ITO substrate for 0 hour (black), 1 hout (red), and 2 hours (green) later the exposure of sun light.

ZnO thin films were used as catalyst for reaction Cr(VI) into Cr(III). From our results UV absorption intensity at 375 nm peak of ZnO/ITO.

CONCLUSION

ZnO nanoparticles have been prepared by using hydrothermal method.

XRD measurement indicate that the prepared ZnO nanoparticles have a crystallite structure which is a characteristic band for the hexagonal structure.

The mean particle size was determined with ~ 96nm for both of TEM and PCCS analysis.

ZnO thin film on ITO substrate was observed with high catalytic activity.

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