



IN APPLIED SCIENCE & ENGINEERING TECHNOLOGY

Volume: 6 Issue: IV Month of publication: April 2018

DOI: http://doi.org/10.22214/ijraset.2018.4009

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Synthesis and Characterization of ZnO Nanoparticles for Hydrophobic Applications

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Abstract: ZnO nanoparticles were synthesized by Co-precipitation method using Zinc Nitrate as precursor and Potassium hydroxide as reducing agent. Characterization was done using UV-Vis spectrophotometer, FT-IR, SEM, XRD and EDX analysis. The XRD study reveals that synthesized ZnO nanoparticles have wurtzite structure. The SEM investigation reveals that surface morphology of ZnO nanoparticles is hexogonal. Hydrophobic spray was prepared by using ZnO nanoparticles and stearic acid as precursors. This hydrophobic spray was tested by coating it on to the cotton cloth and allowed to dry. After drying water was poured on to the cloth and hydrophobic nature was tested.

I. INTRODUCTION

In environment, some plants have (lotus leaves) properties which help to keep the surfaces always clean. Not only for plants but also for insects (butterfly) and bird feathers (penguin) which have water repellency effect (hydrophobicity). This hydrophobicity is derived from nature or biology called Biomimetics. There has been great demand for modifying surface from normal to hydrophobic nature. This hydrophobic nature can be obtained well by placing the nano sized ZnO particles on to required surface [1,2]. There has been a great intrest to investigate the hydrophobic property [3,4]. These have desirable properties to develop nanodevices and nanomaterials, which exhibits excellent hydrophobicity. The hydrophobicity of surface depends on surface microstructure, roughness of surface[5-10] contact angle and rolling angle causing water to bounce and roll-off the surface. The angle formed by the solid surface and the tangent of the droplet is called a *contact angle [11-13]*. The aim of this paper is to prepare the hydrophobic surfaces that have water contact angles larger than 90° have been obtained by controlling the surface topography of expensive hydrophobic materials by various processing methods, such as machining and etching (14-24). By using ZnO nanoparticles and stearic acid hydrophobic spray can be prepared.

II. EXPERIMENTAL DETAILS

A. Preparation of ZnO nanoparticles



Fig. 1. Method of preparation of ZnO nanoparticles



International Journal for Research in Applied Science & Engineering Technology (IJRASET)

ISSN: 2321-9653; IC Value: 45.98; SJ Impact Factor: 6.887 Volume 6 Issue IV, April 2018- Available at www.ijraset.com

ZnO nanoparticles were synthesized by direct precipitation method using zinc nitrate and KOH as precursors. Precipitation occurs through a chemical reaction that forms an insoluble compound out of two or more soluble compounds. Precipitation is the process of solid substance from solution, either by altering the the substance to an insoluble form or by altering the solvent composition to lessen the solubility of substance in it. Precipitation reaction occurs when cations and anions of aqueous solution combine to form an insoluble ionic solid called a precipitate. When the reaction occurs in a liquid, the solid formed is called precipitate. This process is called Co-Precipitation process. In this work, the aqueous solution (0.2 M) of zinc nitrate [Zn(NO₃)₂.6H₂O] and the solution (0.4 M) of KOH were prepared with deionized water, respectively. The KOH solution was slowly added into zinc nitrate solution at room temperature under vigorous stirring, which resulted in the formation of a white suspension. The white product was centrifuged at 5000 rpm for 20 min and washed three times with distilled water, and washed with absolute alcohol at last. The obtained product was calcined at 500 °C in air atmosphere for 3 hr.

B. Preparation of Hydrophobic Spray by ZnO Nanoparticles

0.1M of ZnO was dispersed in ethanol and ultrasonicated for 15 minutes. To this 0.2M of stearic acid dissolved in ethanol was added. The mixture was continuously mixed by using a magnetic stirring for 30 minutes. In this method hydrophobic Zinc Stearate is formed. Zinc stearate will be sprayed and allowed to dried for 24 hours. This modified coatings can be written as $ZnO+ 2CH_3(CH_2)_{16}COOP$ $Zn[CH_3(CH_2)_{16}COOP]_2+H_2O$



(Zinc Stearate)

Fig. 2. Preparation of hydrophobic spray by ZnO nanoparticles

Fig 3 shows stirring of ZnO nanoparticles and stearic acid. The Fig 4 shows prepared hydrophobic zinc sterate.



Fig 3. Stirring ZnO nanoparticles and stearic acid



International Journal for Research in Applied Science & Engineering Technology (IJRASET)

ISSN: 2321-9653; IC Value: 45.98; SJ Impact Factor: 6.887 Volume 6 Issue IV, April 2018- Available at www.ijraset.com



Fig 4. Zinc sterate

III. RESULTS AND DISCUSSIONS

A. X-ray Diffraction (XRD) analysis

The obtained product sample was analyzed to investigate the crystalline nature of synthesized particles. The X-ray diffractogram (XRD) pattern obtained is shown in figure5. The diffractogram pattern were indexed properly for all crystalline peaks and compared with JCPDS data file. Figure shows the major peaks at 2 theta values of 32.645° and 37.979° corresponds to planes of (100) and (101). The estimated crystallite size of ZnO nanoparticles is 66.13nm from Debye-Scherer formula indicating that structure is wurtzite.



Fig 5. XRD analysis of ZnO nanoparticles

The size of ZnO nanoparticles measured by using Debye scherrer equation $t = 0.9 \lambda/(\beta \cos \theta)$

Where, t = Crystallite Size

- β = Full width at half maxima
- $\theta = Bragg's angle$
- λ = wave length of X- rays



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B. Fourier Transform Infrared Spectroscopic (FTIR) Analysis



Fig 6. FTIR spectra of ZnO nanoparticles

Fig. 6 shows the IR spectra of ZnO nanoparticles recorded in the range of $500 - 4000 \text{ cm}^{-1}$. By using the FTIR we can find out the functional groups present in sample. Functional groups are present as following. A strong absorption band were observed at 660.89cm-1 cm⁻¹,1418.84 cm⁻¹, 1568 cm⁻¹, and 3534.65 cm⁻¹ refers to Zn-O stretching, bending of O-H group, stretching vibration of C-O, stretching vibration of O-H respectively.

C. Scanning electron Microscopy (SEM) Analysis



Fig 7. SEM analysis of ZnO nanoparticles

SEM was used to study the morphological analysis of the ZnO nanoparticles. SEM image of ZnO nanoparticles produced by precipitation method is shown in figure 7. From SEM image we found that Shape of the ZnO nanoparticles is hexagonal.

D. UV-Vis Spectroscopy



Fig 8. UV-Vis spectrum analysis of ZnO nanoparticles



Fig. 8 shows the absorbance spectrum of ZnO nanoparticles. It can be clearly seen that the surface Plasmon resonance (SPR) of nickel nanoparticles entered at 386 nm with an absorbance of 1.224.

E. Energy Dispersive X-ray (EDX) Analysis



Fig 9 EDX analysis of ZnO nanoparticles

From the figure 9 we can observed Presence of Zn (Zinc) and O (Oxygen) confirms ZnO (Zinc Oxide) nanoparticles.

F. Hydrophobic Spray on Cloth



Fig 10(a) Cloth before Zinc sterate spray Fig 10(b) Water test on the Cloth before Zinc sterate spray

Initially the cotton cloth was considered without hydrophobic zinc sterate spray as shown in fig10(a) and 10(b). The test was done by 2 or 3 drops of water pouring on to the cotton cloth. By the real nature of the cloth itt it absorbs the water drop totally as shown in fig 10(b).



Fig 10(c) Cloth after Zinc sterate spray Fig 10(d) Water test on the Cloth after Zinc sterate spray



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The prepared Zinc stearate hydrophobic spray was tested on a cotton cloth as shown in the figures 10(c) and 10(d). The Zinc stearate was sprayed on to the cloth and allowed it to dry for 24 hours at room temperature. Now the drop of water is poured on to the cloth and observed . Then the water drop will not be absorb by the cloth. The cloth will repell the water completely. The drop floats on the cotton cloth.

Hence one of the application of Zno nanoparticles is tested by preparing the hydrophobic spray.

IV. CONCLUSIONS

ZnO nanoparticles synthesized by using precipitation method. XRD shows the major peaks at 2 theta values of 32.645° and 37.979° corresponds to planes of (100) and (101). crystallite size of ZnO nanoparticles is 66.13 nm and structure is hexagonal wurtzite. SEM image gives Shape of the ZnO nanoparticles is hexagonal. UV-Vis characterization is done for ZnO nanoparticles and absorbance peak was observed at 386 nm. From FTIR strong absorption bands were observed at 660.89cm-1 cm⁻¹, 1418.84 cm⁻¹, 1568 cm⁻¹, and 3534.65 cm⁻¹ refers to Zn-O, O-H, C-O, O-H. From EDX, the presence of Zn (Zinc) and O (Oxygen) confirms ZnO (Zinc Oxide) nanoparticles. The nature of Zinc Stearate is tested by water after spraying it on to a cotton cloth and allowed it to dry for 24 hours and it was observed the advantage that the droplets were floating and acting as hydrophobic property. In this way we can conclude than by using the ZnO nanoparticles hydrophobic spray can be prepared. No biological affects will be on cotton cloth before and after the Zinc sterate spray.

V. ACKNOWLEDGMENT

This research work was supported by the Chemical department, JNTUA Anantapuramu. I thank Lt. S.Sharada for the guidance and I thank the Riper engineering college for SEM results.

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