International Journal of Advance Research in Science and Engineering
Vol. No.6, Issue No. 01 , January 2017
www.ijarse.com
IJARSE
ISSN (O) 2319 - 8354
ISSN (P) 2319 - 8346

SYNTHESIS OF ZnO NANOPARTICLES FOR ORGANIC POLLUTANT DEGRADATION

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ABSTRACT

In this research work, we have synthesized an effective water filtration system based on the photocatalytic performance of semiconducting dense nanoparticles under natural sunlight. The present study, focus on the photocatalytic activity of nanostructures semiconductor like zinc oxide (ZnO) was prepared by sol-gel methods on the degradation of organic dye such as methylene blue that was investigated. Synthesized ZnO nanoparticles are characterized and study of morphology structure and stability of the synthesized ZnO nanoparticles were studied using scanning electron microscope (FESEM), UV-spectro photometer and Fourier Transform Infrared (FTIR) spectroscopy. The results depicted that the synthesized nanoparticles are moderately stable, roughly spherical with maximum particles in size range within 40-50 nm in diameter.

Key Words: Organic pollutant FTIR, XRD, FESEM etc.

I INTRODUCTION

Environmental contaminants similar to dyes, pesticides and heavy metals in water require remediation and probable removal to make the water it for human consumption. A lot of research is established to purify water from its contaminants, a most important portion of which are organic in nature. Further, the photocatalytic degradation of organic pollutants from water using semiconducting materials has attracted a lot of attention. Such semiconductors are increasingly used for oxidation or degradation of organic dyes and other contaminants particularly in industrial wastewater. The basic mechanism for this remediation is primarily based on the oxygen defects on the surface of the semiconducting materials which when activated by photon irradiation are used to destroy the organic contaminants. The various advantages that the process has are (a) the photocatalytic reaction is not specific to compounds and, therefore, is capable of destroying a spectrum of organic chemicals like hydrocarbon fuels, halogenated solvents, surfactants, pesticides and many hazardous organic chemicals,[1,2] (b) this process is very effective mostly owing to the process of its removal, oven achieving a complete degradation, (c) the process is very resistant to toxicity etc., (d) the process can be applied equally well to liquid (e.g. wastewater and contaminated groundwater) and gaseous streams (e.g. VOC emission), and finally (e) there is a potential to utilize solar energy as reported in this particular work.[3–5] Most photo degradation is still accomplished by exposure to UV radiation although UV sources consume considerable amount of energy.

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In the past two decades, zinc oxide has paying much attention with respect to the degradation of different pollutants due to its high stability, photosensitivity and wide band gap. Most researchers have earlier used ZnO in a nano-powder form dispersed in industrial water for the photo-degradation of organic pollutants. Further, it has been reported that ZnO has a higher photocatalytic efficiency compared to TiO2 in the degradation of several organic contaminants in both acidic and basic media, which has attracted researchers to explore the properties of zinc oxide in many photocatalytic reactions.[6-8] Zinc oxide is consideration to be as a low cost alternative photocatalyst to TiO_2 for degradation of organics in aqueous solutions. The dispersion and surface area of zinc oxide, which depend on the synthesis method, are significant factors for determining its photocatalytic activity for pollutant degradation. Zinc oxide NPs can be synthesis by various type of methods, such as, alkali precipitation, hydrothermal synthesis, thermal decomposition, spray pyrolysis and microwave irradiation, organo-zinc hydrolysis, plasma heat-decomposing, etc. Composites of zinc oxide and silica nanoparticle could be created through coprecipitation route; in this case, the zinc oxide could be covered on the silica nanoparticle surface[9].Photocatalyst is besides called photochemical catalyst and the function is parallel to the chlorophyll in the photosynthesis. In a photocatalytic scheme, photo-induced molecular transformation or reaction takes place at the surface of catalyst. A basic photocatalytic reaction on the generation of electron–hole pair and its destination is as follows: when a photocatalyst is illuminated by the light stronger than its band gap energy, electron–hole pairs diffuse out to the surface of photocatalyst and participates in the chemical reaction with electron donor and acceptor. Those free electrons and holes transform the neighboring oxygen or water molecules into OH free radicals with super strong oxidization. It can oxygenolyse various kinds of organic compounds and some parts of minerals. It may also deoxidize harmful substances like formaldehyde, benzene and ammonia into CO_2 and water free of toxic, harm, and odor. Therefore, photocatalyst may kill viruses, germs, pollen, epiphytes and the like and may decompose formaldehyde, benzenes, ammonia, and other harmful gases, and it will not bring secondary environmental pollution [10]. The degradation of the pollutants catalyzed by ZnO has been studied broadly. ZnO is known of the important photocatalysts because of its unique advantages, high photocatalytic activity, such as its low price and nontoxicity etc.[11, 12]

II MATERIALS AND METHODS

2.1 Materials

Zinc nitrate and citric acid were purchased from Sigma- Aldrich and were all used without further purification and Double distilled water was used as the solvent during this process.

2.2 Synthesis of Zinc Oxide Nanoparticles

ZnO NPs were prepared by the sol-gel method. Firstly from Sigma- Aldrich of zinc nitrate and citric acid were selected as original materials without any purification. 0.5837 g citric acid and 0.826 g zinc nitrate were dissolved into DIW water. In which citric acid acted as both stabilizer and mineralizer. After stirring for 1h, the mixture solution aged in the next 14 h at near room temperature. Then the mixture solution was evaporated and concentrated until changing to wet-gel at 90℃ water-bath. Then the wet-gel was put into drying oven at 120℃

IJARSE
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for 6h, and then the formed mixture was calcined at 500℃ for 3h, and the ZnO nanospheres material was achieved for further characterization.

2.3 Optical Properties

UV-Visible spectroscopy (UV-Vis) refers to absorption spectroscopy in the UV-Visible spectral region. That means it uses beam in the visible and adjacent (near-UV and near-infrared (NIR)) ranges. . In this time uv-vis absorption spectra was takened using a (LABINDIA Uv- Visible 3000+) spectrophotometer where the cuvette path length was set to 1.0 cm. The particles were dissolved in DIW water, and solution was placed in a quarts cuvettes. The absorption in the visible range openly affects the perceived colour of the chemicals involved. In this region of the electromagnetic spectrum generate by the electronic transitions.

2.4. Powder X-Ray Diffraction

XRD patterns of the powdered samples were obtained on a Phillips X'Pert materials research diffractometer using secondary monochromated Cu Kα radiation (λ = 1.54060 A°) at 40 Kv/50mA. Samples were supported on a glass slide. Measurements were taken using a glancing angle of incidence detector at an angle of 2 for 2θ values over 10–70 in steps of 0.05 with a scan speed of 0.012.

2.5. FESEM analysis

The FESEM image was taken with very high resolution and MATLAB analysis gives the pixel depth of the image equal to 24bits and the image format as JPEG. The FESEM Images have been taken from MNIT Jaipur, Rajasthan, India.

2.6. Fourier Transform Infrared (FTIR) Spectroscopy

FTIR analysis range 4000 to 400 cm^{-1} using Bruker -Tensor Spectrum in the diffuse reflectance mode at a resolution of 4cm^{-1} in KBr pellets. The powder sample was placed on a sample holder and the spectrum was recorded.

III RESULTS AND DISCUSSION

3.1 UV-Visible Analysis

UV-Visible absorption spectroscopy is widely used technique to examine the optical properties of nano sized particles. The prepared zinc oxide white crystalline powder was not soluble in water and almost in all organic solvents. ZnO nano particles UV-Visible spectra recorded by dispersed in methanol solution and sonicated for 5 to 10 min. Fig. 4 shows the absorption spectroscopy of the ZnO nanoparticles in the UV-spectral region. ZnO exhibits a sharp band at 354 nm, which corresponds to the formation of ZnO nanoparticles.

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ISSN (O) 2319 - 8354
ISSN (P) 2319 - 8346

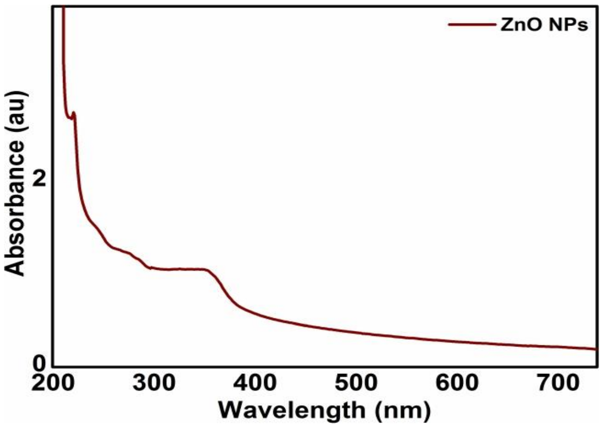

Fig.1. UV-Vis. spectra of ZnO NPs synthesized by sol-gel method

3.2 XRD Analysis

The crystal structures of the synthesized samples were determined by powder X-ray diffraction (XRD) using a copper Kα radiation source at 40 kV and 200 mA in steps of 0.02. Data were recorded ranging from 10° to 70°. Fig. 2 showed the XRD patterns of as-synthesized samples obtained by sol-gel method whose reflection peaks can be readily indexed with the wurtzite ZnO (JCPDS No. 79-2205). Prepared ZnO nanospheres were obtained by the sol-gel method, the crystal structure of ZnO was shown in Fig. 3, which belonged to the wurtzite structure. It can be seen that lattice parameters were a=0.3249 nm and c=0.5206 nm.

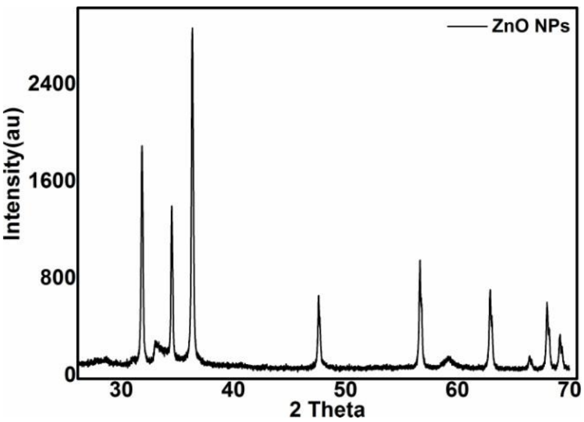

Fig.2. XRD spectra of ZnO synthesized by: sol-gel method

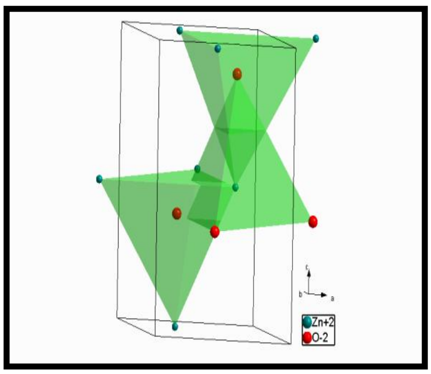

Fig.3. Crystal structure of ZnO

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ISSN (O) 2319 - 8354
ISSN (P) 2319 - 8346

3.3 Morphology Analysis:

SEM images of the products were shown in Fig. 4. ZnO NPs was prepared by sol-gel method calcined at 500℃ for 3 hours and pH was 8. SEM image showed relatively more uniform ZnO NPs prepared by sol-gel method look like nanospheres, with diameter range 10-50nm.

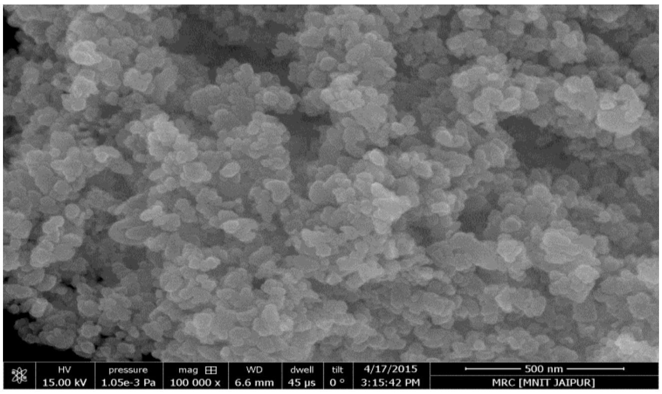

Fig.4. SEM image of ZnO NPs produced by the Sol-gel method

3.4 FTIR Analysis

FT-IR spectrum of ZnO nanoparticles (Fig. 5) showed significant absorption peaks at 573 and 1577, 3372cm^{-1}. The absorption band at 573 cm^{-1} due to Zn-O stretching vibration mode. The weak band near 1577 cm^{-1} is assigned to H-O-H bending vibration mode were presented due to the adsorption of moisture, when FTIR sample disks were ready in an open air atmosphere. These explanations provided the evidence for the presence of hydration in the structure and intense broad band near 3372 cm^{-1} represents the hydrogen bonded O-H stretching vibration mode.

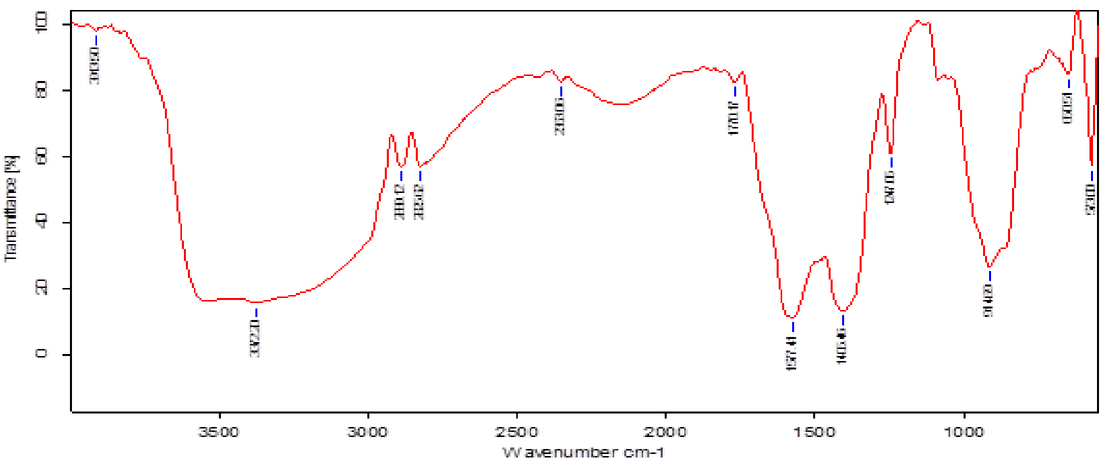

Fig.5 FTIR spectra of synthesized ZnO NPs

3.6 Photocatalytic activity of ZnO NPs

Photocatalytic activity of ZnONPs was investigated by measuring the photocatalytic degradation of methylene blue in water under the illumination of UV light. The ZnO nanoparticles of 50 nm in common particle size were chosen for the evaluation of photocatalytic activity. As shown in Fig. 6, it was found that the ZnO nanoparticles were effective on the degradation of the methylene blue. Higher photocatalytic activity of the ZnO nanoparticles is considered due to the higher surface area of the ZnO nanoparticles. At higher surface area, larger contact area between photocatalyst and target material can be obtained. It also meant that higher degree of UV light absorption could occur at the smaller particle size in the test solution.

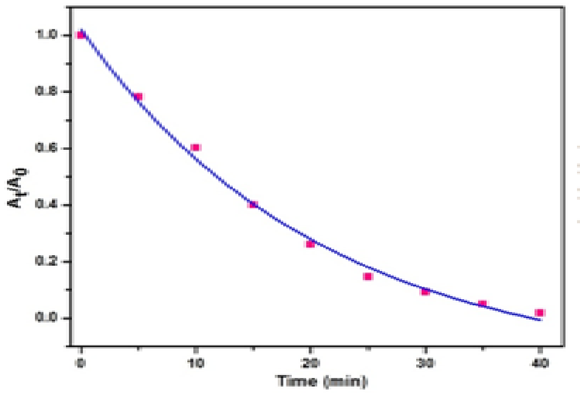

Fig.6. Effect of catalyst loading on the photodegradation of methylene blue using ZnO nanoparticles.

lV. CONCLUSION

In this present study, ZnO NPs have been successfully synthesized using Zinc nitrate and citric acid solution as precursors via sol-gel approach. By carefully controlling the process window, various morphologies of ZnO particles such as disk like and spherical NPs could be produced. The crystallite size calculated from the XRD is 50 nm. FT-IR results confirm that the presence of Zn-O at 573cm^{-1} as well as UV-visible Spectroscopy the absorption spectrum was 374 nm absorbed. The photo degradation of methylene blue using ZnO spherical nanoparticles process was investigated. In which catalysts may further enhance the photocatalytic activity due to its high surface to volume ration which will facilitate the better adsorption of dyes.

V. ACKNOWLEDGMENT

The authors are sincerely thankful to the Department of Science and Technology and their sub wing Science and Engineering Research Board of the, India (SB/S1/PC- 31/2012) for their financial support.NCL, Pune and IIT Rorkee, India, are also thanked by the authors for providing XRD and FESEM facilities, respectively.

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