

Microwave Assisted Fast Synthesis of Flower-like ZnO Based Guanidinium Template for Photodegradation of Azo Dye CongoRed

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Abstract—ZnO nanostructure were synthesized via microwave method using zinc acetate as starting material, guanidinium as structure directing agents, and water as solvent. This work investigates the photodegradation of azo dyes using the ZnO Flower-like in aqueous solutions. As synthesized ZnO samples were characterized using X-Ray powder diffraction (XRD), scanning electron microscopy (SEM), and FTIR spectroscopy. In this work photodecolorization of congoed azo dye under UV irradiation by nano ZnO was studied.

Keywords—Photo catalyst, Nano crystals, Zinc Oxide

I. INTRODUCTION

DYE pollutants are harmful species produced by industries and their removal is of importance from environmental point of view. Release of dye containing waste water in the ecosystem leads to huge aesthetic pollution and perturbations in aquatic life [1, 2]. Most of the organic dyes are not easily degradable by standard biological methods. Recently photocatalytic degradation of various kind of organic and inorganic pollutant has been extensively studied using semiconductor metal oxides as photocatalysts [3, 4]. ZnO is a wide band gap oxide semiconductor with a direct energy band gap of about 3.3 eV, and consequently absorbs UV radiation through bond to bond transition [5, 6]. In this study, ZnO nanorods are successfully prepared via hydrothermal method. Congo red, a typical azo dye with two (-N=N-) azo bonds, is used to investigate the catalyst efficiency for photocatalytic elimination of pollutants. The extent of photodecolorization is found to be 90% in 160 min, a 30 W high pressure Hg Vapor lamp is used as light source.

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II. EXPERIMENTAL SECTION

A. Materials

All chemicals were used without further purification. Congo Red (C.I. Direct Red 28, M.W. = 696.67 g/mol), sodium hydroxide, zinc acetate, Guanidinium, solution of dye were prepared by dissolving amount of dye in distilled water before experiment. The concentration of dye solution was 5 ppm.

B. Synthesis of ZnO Flower-like

In a typical procedure 0.8 g of zinc acetate is dissolved in 40 ml of deionized water under vigorous stirring. A 4M solution of NaOH is then added drop wise until solution is transparent at pH of 12. A 0.1 M aqueous solution of guanidinium carbonate is then added to the above mixture and stirred for 30 min. Finally, the mixture is exposed to microwave irradiation (power 75%) for 2 minutes. A microwave oven with 800 W power was used for irradiation. The mixture is then centrifuged and the white solid product is washed several times with double distilled water and dried at 60°C under vacuum.

C. Photocatalytic reaction studies

To study the photocatalytic reaction, 30 ml of the 5 ppm solution of Congo Red was used as a suspension along with 25 mg of the catalyst in an open Pyrex reaction vessel and irradiated using UV lamps (30 W, UV-C, $\lambda = 253.7$ nm). The suspension was magnetically stirred during the photolysis. Air was blown in to the reaction by an air pump to maintain the solution saturated with oxygen during the reaction. During photocatalytic reaction, the temperature of the dispersions was maintained at 35 °C. First dye solution was stirred in the dark for 30 min. Then the suspension was irradiated by UV lamp and Three milliliters of the homogenized dispersion aliquots were taken at regular intervals, starting from zero time and centrifuged at 3500 rpm for 10 min. The reaction was monitored as a function of illumination time by measuring the absorbance of Congo Red. (UV-Visible spectrometer (Shimadzu UV 2100)).

III. RESULTS AND DISCUSSION

Morphology of flower-like zinc oxides is recorded using scanning electron microscopy (Fig.1). Flower-like ZnO structures are about 2 μ m and consist of well-defined petals with average size of about 600-700 nm in length, 300-400 nm

in width, and 50-70 nm in tip. The XRD pattern is shown in fig .2. All diffraction peaks are indicate to the hexagonal phase with wurtzite structure (JCPDS card no.36-1451 with unit cell parameter $a=b=3.24\text{\AA}$ and $c=5.21\text{\AA}$). The FTIR spectrum of ZnO is shown in fig .4. A strong band appeared 600 cm^{-1} is attributed to the Zn-O stretching band. Fig.3. shows normalized absorbance at 497, 235 and 347 nm which are plotted as function of Irradiation time. After adding ZnO nano rods in dark for 30 min the absorbance decrease because the adsorption of the dye on the ZnO surface. The decrease of the absorbance is a bout 20%. The mechanism has been recognized that hydroxyl radical $\text{OH}\cdot$ acts as reagent for the mineralization of organic pollutants. Electrons in the conduction band on the catalyst can reduce molecular oxygen to form superoxid anion also conductive electrons are responsible for the production of hydroxyl radical.

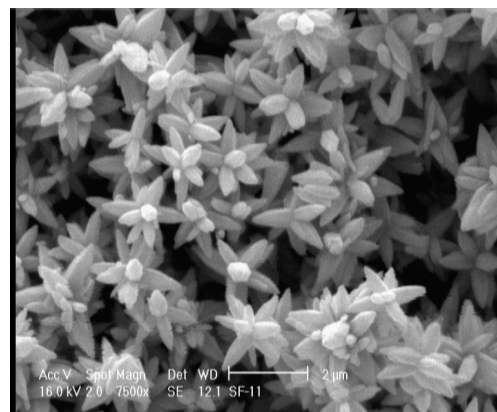
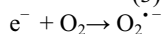
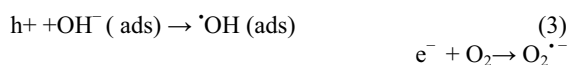
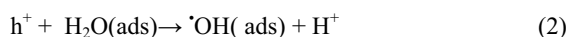


Fig.1. SEM image of ZnO Flower-like



$\cdot\text{OH} + \text{Dye}$

degradation of the Dye

UV light illumination leads to decrease absorption bands of the dye in the visible region to disappear completely.

The photo degradation efficiency of Congo Red was defined as follows:

$$\text{Photo degradation efficiency (\%)} = \frac{C_0 - C_t}{C_0} \times 100$$

IV. CONCLUSION

In summary, Azo dyes such as Congo Red are the ideal condition for the elimination of $\text{N}=\text{N}$ azo group containing pollutants. The hydrothermal method has been successfully used for synthesis of ZnO nano Rods with hexagonal structure. The method is simple, environmentally friendly and low cost for production ZnO photocatalyst. The results obtained in this study demonstrate that ZnO nano Rods was a very promising photocatalyst for decolorization of Congo Red azo dye. The extent of photo decolorization is found to be 90% in 160 min.

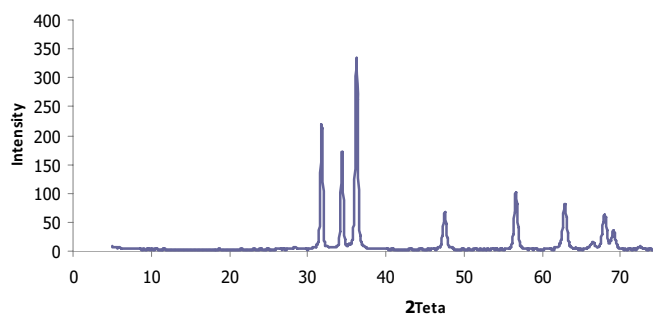


Fig.2. XRD pattern of as prepared ZnO

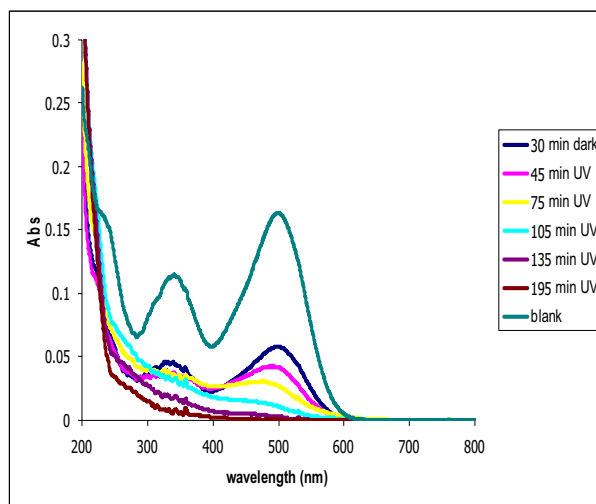


Fig.3. The UV-Vis spectra of Congored in aqueous at different irradiation time

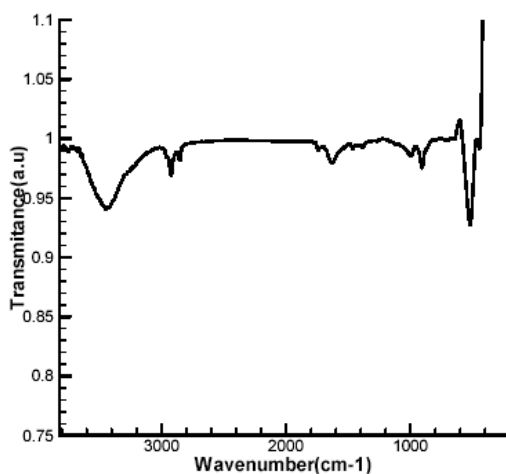


Fig.4 FTIR Spectrum of ZnO photo catalyst

REFERENCES

- [1] Shao, X. Wang, Q. Fan "Photocatalytic reduction of Cr(VI) to Cr(III) in solution containing ZnO or ZSM-5 zeolite using oxalate as model organic compound in environment" *Microporous and Mesoporous Materials*, 2009, pp 243–248.
- [2] C. Hariharan, "Photocatalytic degradation of organic contaminants in water by ZnO nanoparticles" *Applied Catalysis A: General*, 2006, pp 55–61.
- [3] M. Mrowetz, E. Selli, "Photocatalytic degradation of formic and benzoic acids and hydrogen peroxide evolution in TiO₂ and ZnO water suspensions" *Journal of Photochemistry and Photobiology A: Chemistry*, 2006, 15-22.
- [4] F. Chen, Y. Xie, J. Zhao, G. Lu, "Photo catalytic degradation of dyes on a magnetically separated photocatalyst under Visible and UV irradiation" *Chemosphere*, 2001, pp 1159-1168.
- [5] C.C. Lin, Y.Y. Li, "Synthesis of ZnO nanowires by thermal decomposition of zinc acetate dihydrate" *Materials Chemistry and Physics*, 2009, pp 334-337.
- [6] M. S. Niasari, N. Mir, F. Davar, "ZnO nanotriangles: Synthesis, characterization and optical properties" *Journal of Alloys and Compounds*, 2009, pp 908-912.