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Synthesis, characterization and investigation photocatalytic degradation of Nitro Phenol with nano ZnO and ZrO_2

N. Assi[1, 2, *]
A. A. Mehrdad Sharif[2]
Q. S. Manuchehri Naeini[2]

[1]*Department of Chemistry, Science and Research Branch, Islamic Azad University, Tehran, Iran.*
[2]*Department of Chemistry, Faculty of Chemistry, North Tehran Branch, Islamic Azad University, Tehran, Iran.*

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ABSTRACT

ZnO and ZrO_2 photo catalysts were synthesized by sol-gel auto combustion method. The products were characterized by X-ray Diffraction (XRD) and Scanning Electron Microscopy (SEM). Structural and morphological properties of nano particles were investigated and the average crystalline size of ZnO and ZrO_2 was obtained 44 and 51 nm, respectively. Also, photo catalytic degradation of Nitro phenol from aqueous solution by using nano scale ZnO and ZrO_2 powders under UV-C light irradiation was studied and degradation of Nitro phenol was 98% for ZnO in 40 minutes and 83% in 100 min for ZrO_2.

Keywords: *Nitro phenol; Sol-gel auto combustion method; ZnO; ZrO_2; Photo catalytic; Degradation.*

INTRODUCTION

Nitro phenol is one of the important contaminating materials that have high toxicity level receiving in water resources. Its high toxicity, even at low concentrations, has motivated the search and improvement of many treatment techniques. In this sense, photo catalysis can yield feasible, convenient methods for the treatment of phenolic wastewaters [1-3]. The photo catalytic degradation process is one of the developing technologies in the treatment of wastes containing persistent organic materials. The photo catalysis system using semi conductive particles is a very promising technique and has attracted extensive attention in recent years [4]. Semiconductor nano particles have attracted the interest of many academic and industrial researchers because of their properties based on quantum size effects and high surface area [5, 6]. Among these materials, Zirconia attracts attention due to its application as an engineering ceramic for both mechanical and electrical purposes. ZrO_2 is a wide band gap semiconductor (~5.0 eV) and ZnO (~3.4 eV) has been proven to have the photo catalytic performance. Photo catalyst was synthesized with different process such as co-precipitation, [7, 8] sol-gel, [9, 10] and Chemical Vapor Deposition (CVD) [11].

* Corresponding author:
Navid Assi
Department of Chemistry, Science and Research Branch, Islamic Azad University, Tehran, Iran.
Tel +98 9122060649
Fax +98 21 22977853
Email navid_a30@yahoo.com

It is well recognized that sol-gel auto combustion is an effective method for synthesis of semiconductor nano particles for different advanced applications such as photo catalysis. Sol–gel auto-combustion is a way with a unique combination of the chemical sol–gel process and the combustion process based on the gelling and subsequent combustion of an aqueous solution containing salts of the desired metals and some organic fuel, giving a voluminous and fluffy product with large surface area. This process is less complicated than the others [12, 13]. In this study, we synthesis ZnO and ZrO_2 with sol-gel auto combustion method and investigate photo catalytic degradation of Nitro phenol in aqueous solution.

EXPERIMENTAL

Materials

In this method, Nitro Phenol [$C_6H_5NO_3$], Zink nitrate [Zn $(NO_3)_2.6H_2O$)], Zirconium nitrate [Zr $(NO_3)_2.6H_2O$], Glycine [$C_2H_5NO_2$], Ammonia 25% were obtained from Merck Co. (Germany). All material was analytical grade and use without any purification. Deionized water was used for the preparation of all the samples.

General synthesis of nano ZnO and ZrO_2

In this step, nano particle synthesis by the method describe via Mehrdad Sharif et al [14]. The mole ratio of Zr $(NO_3)_2.6H_2O$ to Glycine, was fixed in 1:3. Materials were dissolved in a minimum amount of deionized water to get a clear solution. Ammonia solution (25%) was slowly added to adjust pH to 7. The solution was allowed to evaporate on a hot plate and maintained at 60 ˚C under continuous stirring. After the evaporation of water, the resulting gel was ignited to form a loose powder. Finally the precursor was calcined at 600 ˚C for 2 hours to obtain ZrO_2 nano particles. All the above steps were repeated for preparing the ZnO nano photo catalyst.

Appraisement of the photo catalytic activities of ZnO and ZrO_2 nano particles

In order to show their potential environmental application, the degradation of Nitro Phenol was evaluated. First, the solution of 3 mg/L Nitro Phenol was prepared in deionized water and then 100 ml of prepared solution transfers in a purely clean container. An amount of 0.04 mg of nano photo catalyst was added to containers. Solutions were placed at a distance of 30 cm from UV-C lamp with power of 30 W for a time interval of 100 minutes. The solutions were continuously stirred throughout the whole time. Samples were taken from the solution and were filtered and centrifuged for 20 minutes to remove all nano particles completely. Finally, clear transparent solutions were obtained. Afterwards, samples were taken for obtaining solutions and put in the double beam spectrophotometer to measure the absorbance of remaining Nitro Phenol. This process applies for determination ZnO and ZrO_2 nano photo catalyst degradation.

RESULTS AND DISCUSSION

X-ray Diffractions of Nano particles

The phase and purity of nano powders were determined from the XRD patterns Figure 1 (a, b). Well-defined sharp peaks indicate the good crystalline quality and confirm the formation of single-phase ZnO and ZrO_2 nano photo catalysts. The diffraction peaks appeared in the XRD patterns can be indexed with the standard patterns for ZrO_2 (JCPD 01-079-1768) and ZnO (JCPD 01-079-0208). The average crystalline size calculated from Debeye-Scherrer equation (eq.1) was 44 and 51 nm for ZnO and ZrO_2, respectively.

$$D = \frac{0.89\ .\lambda}{\beta.\cos\theta} \qquad (1)$$

D: Average crystalline size (nm)

λ: The X-Ray wavelength

β: The full width at half maximum

θ: Half diffraction angle

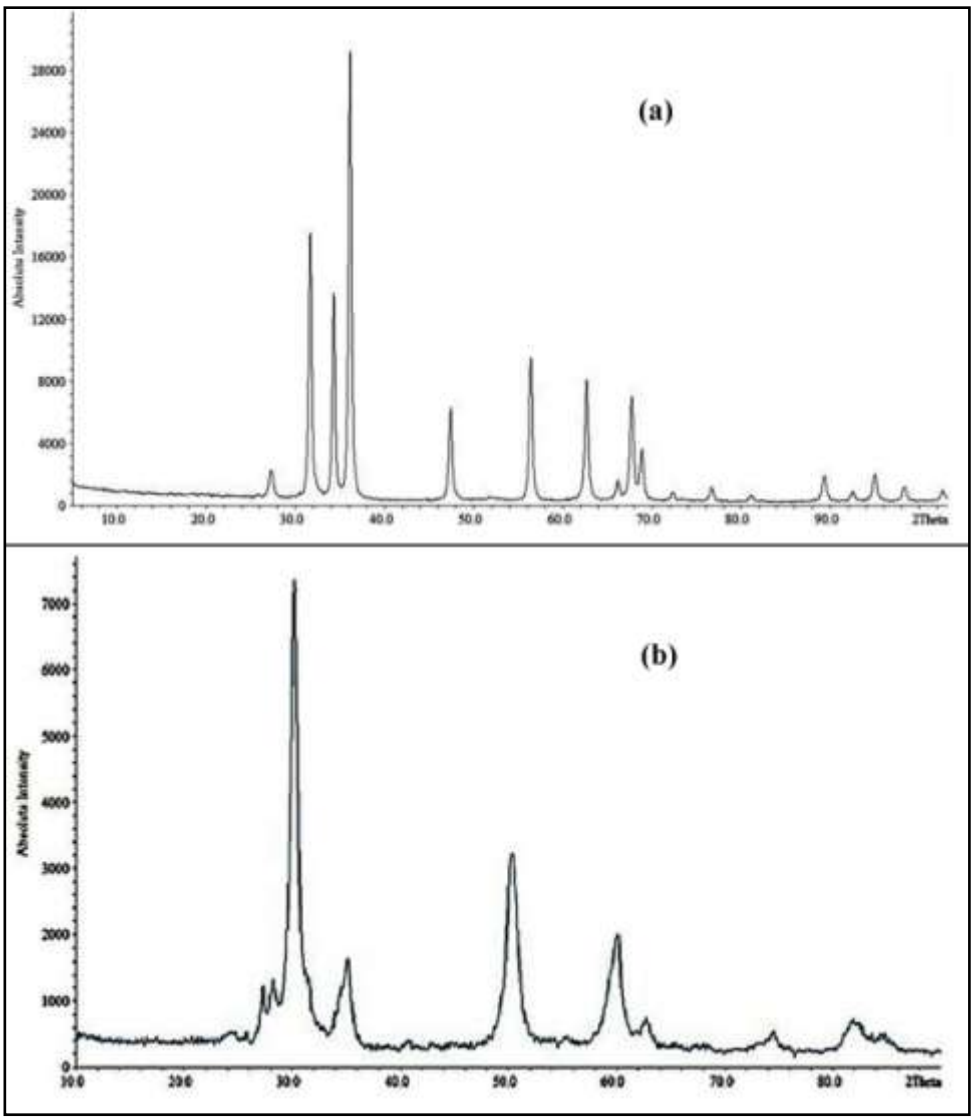

Fig. 1. XRD patterns of nano particles a. ZnO and b. ZrO2.

SEM analysis of ZnO and ZrO_2

SEM images of ZnO and ZrO_2 nano photo catalysts are shown in Figure 2 (a, b). Figure 2a shows that the ZnO nano powder contains spherical nano particles, whereas Figure 2b shows a cavity structure for ZrO_2 nano powders.

Evaluation of Photo catalytic of ZnO and ZrO_2 Nano particles

The solution of 3 mg/L Nitro phenol, with a volume of 100 cc was prepared. Then 0.04 g nano particles added to a solution, and put under the (30 W UV-C) irradiation. Then samples are taken at 10 minutes interval, up to 10 samples. All samples are filtered and each sample located in UV spectroscopy double beam instrument and their absorption reads under Nitro phenol 279 nm.

Degradation of Nitro phenol can be calculated from following equation:

$$X = \frac{A_0 - A}{A_0} \times 100$$

X = Degradation percentage

A_0= Initial absorption

A = Absorption after t minutes

Figure 3 shows degradation of Nitro phenol in presence of nano ZnO and ZrO_2.

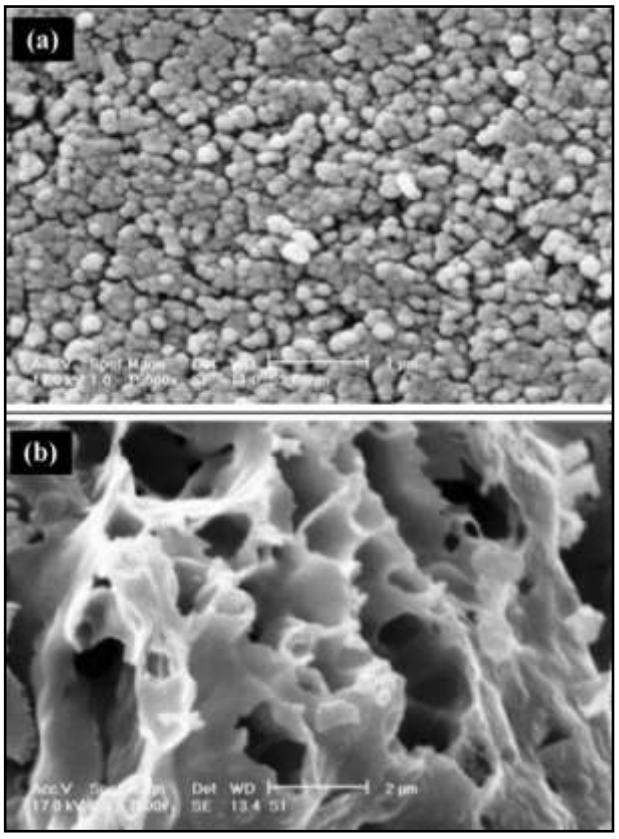

Fig. 2. SEM images of nano particles a. ZnO and b. ZrO_2 nano particles

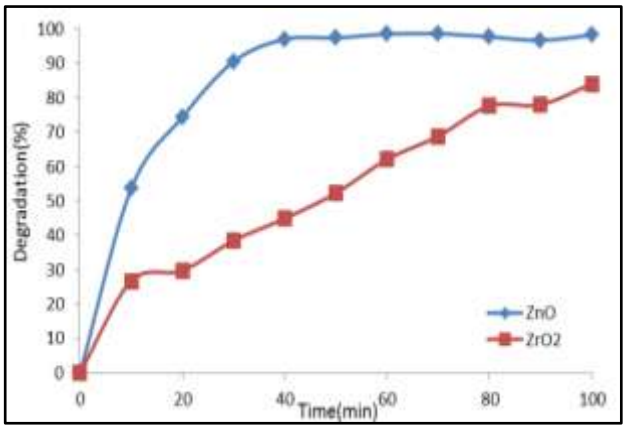

Fig. 3. Degradation of Nitro phenol in presence of ZnO and ZrO_2 nano photo catalyst.

Degradation of Nitro phenol in the presences of nano ZnO photo catalyst after 40 minutes was 98% of total volume, for ZrO_2 after 100 minutes was 76% of total volume, have observed.

CONCLUSIONS

ZnO and ZrO_2 photo catalysts were synthesized by sol-gel auto combustion method. The average crystalline size was determined by the Debeye-Scherrer formula by X-ray spectrum data and estimated about 51 nm to ZrO_2 and 44 nm for ZnO. The observations indicate high photo catalytic degradation strength of nano photo catalyst ZnO over Nitro phenol against ZrO_2. ZnO has a large initial rate of activities and its absorption efficacy of UV-C irradiation.

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