

Synthesis, Characterization, Synergic Adsorption-Photocatalytic Studies of Novel ZnO Nanoparticle

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ABSTRACT

In this study Nano Zinc oxide and Titanium Oxide synthesized using Dextrose as fuel by solution combustion method. Thus obtained powder particle is characterized for their purity, porosity, stability, particle size and stoichiometry by FT-IR, XRD, SEM-EDAX, TEM, PL and UV-VIS DRS Spectrum techniques. The Synthesis of Zinc oxide nanoparticles has high photo catalytic activity. The proposed mechanism for the photocatalytic degradation of Rhodamine Blue dye by ZnO was found to be due to the formation of hydroxyl radicals, which are active species in the photocatalytic reactions. The prepared catalyst was found to be stable and reusable for industrial application.

KEYWORDS: ZnO oxide Nanoparticle, Characterization, Rhodamine Blue dye, Photocatalytic

How to cite this paper: V. Ramya | S. Indhumathi "Synthesis, Characterization, Synergic Adsorption-Photocatalytic Studies of Novel ZnO Nanoparticle" Published in International Journal of Trend in Scientific Research and Development (ijtsrd), ISSN: 2456-6470, Volume-6 | Issue-4, June 2022, pp.1537-1541, URL: www.ijtsrd.com/papers/ijtsrd50302.pdf



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1. INTRODUCTION

1.1. GENERAL INTRODUCTION OF NANOMATERIAL

Zinc oxide (ZnO), semiconductor compounds, is a very versatile and important material. ZnO has a unique position among semiconducting oxides due to its piezoelectric and transparent conducting properties. The obtained powder zinc oxide nanoparticle has high electrical conductivity and optical transmittance in the visible region. These properties make it an ideal candidate for applications like transparent conducting electrodes in flat panel displays and window layers in solar cells. ZnO has a wide band gap (3.37 eV) and a large excite on binding energy (60 meV) and exhibits many potential applications in areas such as laser diodes, solar cells, gas sensors, optoelectronic devices.

A wide band gap has many benefits like enabling high temperature and power operations, reducing electronic noise, raising breakdown voltages. By proper alloying with MgO or CdO, the band gap can be tuned in the range of 3-4 eV. ZnO nanostructures

have an active role to play in nano devices like Nano gas sensors because the huge surface area enhances the gas sensing properties of the sensors.

Bio-safe characteristics of ZnO make it very attractive for biomedical applications. A method for economical mass production of ZnO nanostructures would therefore be very useful. To synthesis Zinc oxide nanostructures.

The most important application areas are paints and varnishes as well as paper and plastics, which account for about 80% of the world's titanium dioxide consumption. Other pigment applications such as printing inks, fibers, rubber, cosmetic products and food account for another 8%. The rest is used in other applications, for instance the production of technical pure titanium, glass and glass ceramics, electrical ceramics, metal patinas, catalysts, electric conductors and chemical intermediates.

2. MATERIALS AND METHODS

2.1. Synthesis

Keeping in view the extensive uses of ZnO various types of synthesis techniques have been formulated over the years. ZnO nanostructures can be synthesized by several methods among these methods solution combustion is the best method. Solution combustion synthesis is a novel technique that has been used successfully for the preparation of ceramic and phosphor materials. This method was successfully employed to prepare LiNiO_2 , LiCoO_2 and LiMnO_4 . The main advantage of this method, compared to the solid state sintering method, is that the experiment is completed within 10 min. The basic principle of the method is the decomposition of an oxidizer, e.g., a metal nitrate, in the presence of a fuel. The fuel gets ignited by the oxidizer to yield oxide materials derived from the metal salts. The fuel used in the present study was dextrose which acts as fuel.

2.2. Materials and Methodology

2.2.1. Chemicals Required

1. Zinc nitrate -10 g
2. Tetraisopropylorthotitanate -10ml
3. Dextrose- 3g

2.2.2. Experiment of ZnO

Zinc oxide was synthesized by the combustion of aqueous solutions containing stoichiometric amounts of the Zinc nitrate (Merck) and Dextrose (Merck). Stoichiometric composition of the redox mixture was calculated based on the total oxidizing and reducing valencies of the oxidizer and the fuel keeping the O/F ratio unity. Zinc nitrate and dextrose were dissolved in 25 ml of water contained in a beaker and placed on a hot plate for 15 minutes as the solution dehydrates to form a disposition like a gel. Then the beaker was placed in a preheated muffle furnace at 400°C . The solution boils, ignites with a flame and the entire reaction was completed within 5 minutes. The powder was highly amorphous, a portion of the sample was calcined at 650°C and the XRD pattern and EDX of this powder confirms the formation of ZnO.

3. RESULT AND DISCUSSION

3.1. FESEM Analysis

Field Emission Scanning electron microscope (FESEM) images of the synthesized ZnO samples using dextrose as a fuel are shown in Figure. FESEM image of the particles were agglomerated and their sizes lie in the range of 35-85 nm. The agglomerated particles neck with their neighbours. They form elongated spindle-like shapes intermitted with voids, ensuring high surface area. The particles of the nano zinc oxide synthesized by combustion method show

greater extent of agglomeration hence no distinct particles were obtained.

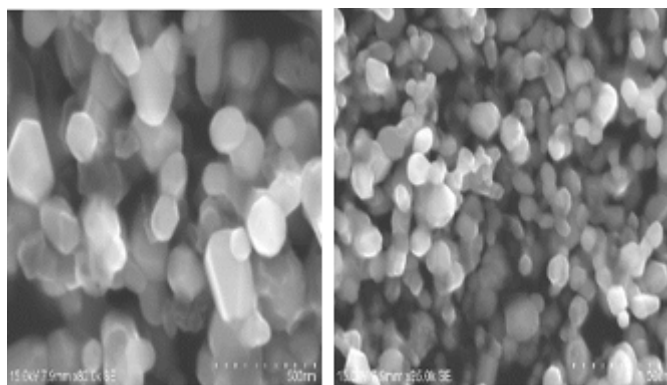


Fig: FESEM images of Zinc oxide

Fig. 3.1. Schematic diagram of photoreactor

4. EDAX Analysis

Energy dispersive X-ray analysis Shows the composition of the compound. It's a semi-quantitative method to determine the composition of the compounds and highly Qualitative for identify the constituent present in the powder. The composition of Zinc oxide prepared by combustion method is given in table and figure

Table 4.1 composition of zinc oxide

Element	Atomic percentage (%)
O K	58.98
Zn L	41.02
Total	100

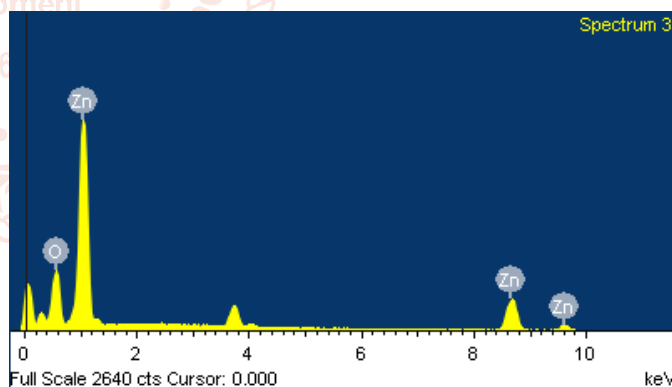
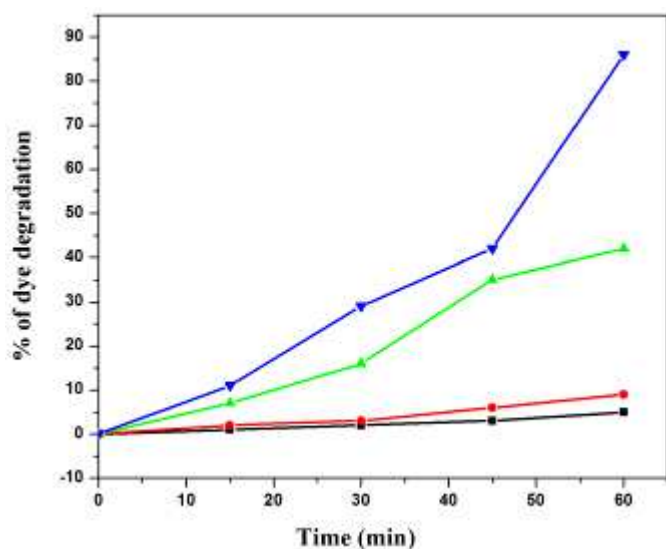


Fig 4.2: EDX spectrum of Zinc oxide

4.1. Photocatalytic application of Rhodamine Blue dye with artificial UV- light irradiation

The Primary analysis of photodegradation of Rhodamine Blue dye in aqueous medium in the presence of synthesized ZnO NPs and the atmospheric air were studied using multi lamp photoreactor with mercury UV- light irradiation at 365 nm. The initial Rhodamine Blue dye concentration 1×10^{-4} M and the pH of dye is neutral. It is shown to the dark in colour. After the photo degradation colour changes at irradiation times 60 min shown in was respectively. The reaction time affords the photo degradation of Rhodamine Blue dye. Synthesized NPs material exhibited much enhanced to photocatalytic activity

when compared to that of Dark and Nil catalyst, powder ZnO and synthesized ZnO NPs. The Rhodamine Blue dye is resistant to photolysis with synthesized ZnO NPs in the dark in dye (1×10^{-4}) concentration was observed Rhodamine Blue dye undergoes % of degradation 0 (0%), 15 (11%), 30 (29%), 45 (42%) and 60 (86%) that of powder ZnO % of degradation 0 (0%), 15 (7%), 30 (16%), 45 (35%) and 60 (42%) under UV-light in 60 min irradiation. synthesized NPs higher photocatalytic activity was obtained it shows in **FIG**

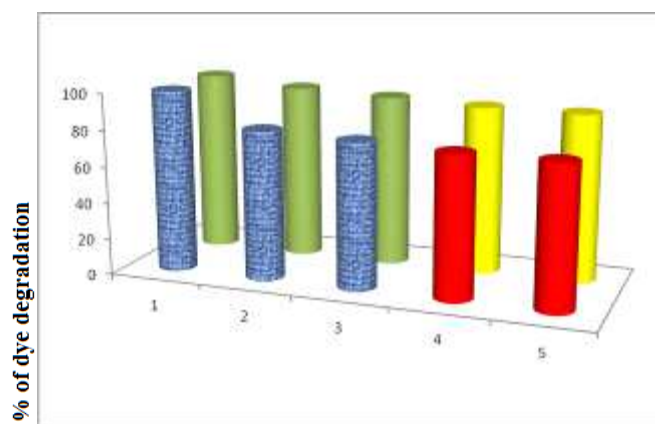


4.2. Stability of the Reusability of synthesized ZnO

The Stability of the Reusability of photocatalyst prepared Prepare ZnO investigated by repeating Rhodamine Blue dye photocatalytic degradation experiments five times. After each cycle, the photocatalysts were washed thoroughly with water, and a fresh solution of Rhodamine Blue dye was made before each photocatalytic run in the photoreactor under UV- light irradiation. The complete degradation occurred in the Prepare ZnO on Rhodamine Blue dye for 4th to 5th at cycles obtained 78 % and 92 % degradation it shows in **Fig.4.12**. The results indicated the prepared catalysts are stable and reusable waste water treatment analysis. After achievement of the degradation process the solution was tested for Zn³⁺ leaching with sodium sulfide. There is no precipitation in black color was formed. As there is no further leaching of this photocatalyst is non-hazardous for wastewater treatment. There is no significant change in reaction, indicating the stability of photocatalyst. This is due to the loss of catalysts, during the water washing of catalysts, which was not observed in the naked eye. Consequently suggests Prepare ZnO have exceptional stability and reusability for photodegradation of wastewater treatment for industrial application [55].

Organic dye + OH^{*} → Mineral acids + CO₂ + H₂O

Organic dye + OH^{*} → Mineral acids + CO₂ + H₂O



1 to 5 cycle, Time (min)

Fig. Stability and Reusability on Rhodamine Blue dye degradation; (a) Chemical synthesis ZnO nanopowder and (b) Synthesis of ZnONPs

5. CONCLUSIONS

In this study nano Zinc oxide and Titanium Oxide synthesized using Dextrose as fuel by solution combustion method. It is well known that this simple combustion method is the easiest route to synthesize semiconductor and it can be used in industries upon scaling up. When comparing with other methods, it is a simple, quick, and inexpensive method involving a single-step reaction. Thus synthesized powder is characterized for their purity, porosity, stability, particle size and stoichiometry by FT-IR, XRD, SEM-EDAX, TEM, PL and UV-vis DRS Spectrum techniques. The crystallite size computed using Scherer equation with XRD profiles and found using SEM and TEM images. Nano zinc oxides could be successfully prepared by solution combustion method, which is a low cost method. The purity of the prepared zinc oxides is quite high. FESEM results suggested the Zinc Oxide prepared from solution combustion method consist of elongated spindle elongated and spherical-like shapes like structure. HR-TEM results suggested the Zinc oxide and Titanium Oxide prepared from solution combustion method consist of nano spherical-shape and tetrahedral shaped structure as shapes like structure. EDAX results showed the presence of only zinc and oxygen. From UV result we confirmed presence of Zinc which get absorbed at 435nm and 377 nm. Peaks observed from FT-IR confirming the presence of Zinc oxide. XRD pattern is compared with JCPDS and the results correlated with zinc oxide the phase structure, which is confirmed as Wurzite. PL spectra confirmed the suppression of recombination of the photogenerated electron-hole pair by synthesized ZnO nanomaterial. A low electron and hole recombination rate implies a lower luminescence emission intensity and higher photocatalytic activity. UV-Vis-DRS demonstrated the

decrease in the direct band gap energy high photocatalytic activity. The photocatalytic activity of the NPs was evaluated by the degradation of Rhodamine Blue dye in aqueous solution under UV-light at 365 nm. The Synthesis of ZnO NPs was high photocatalytic activity. The proposed mechanism for the photocatalytic degradation of Rhodamine Blue dye by ZnO was found to be due to the formation of hydroxyl radicals, which are active species in the photocatalytic reactions. The prepared catalyst was found to be stable and reusable for industrial applications

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