

New Age Photocatalyst for Photodegradation of Industrial Effluents

V.L.CHANDRA BOSS¹, T.LAKSHMIKANDHAN²,

^{1,2}Associate Professor, Department of Chemistry,

BIST, BIHER, Bharath University, Chennai-73

Chandraboss.che@bharathuniv.ac.in, lakshmikandhan.che@bharathuniv.ac.in

Abstract

Nanometal oxide doped silica matrix (ZnO@SiO₂) was synthesized by sol-gel method. The crystal structure and surface morphology of the material were characterized by X-ray diffraction (XRD) and scanning electron microscopy (SEM) with energy dispersive X-ray analysis (EDX). It is found that nanometal oxide is trapped on the pores of the silica matrix. In the application side the synthesized ZnO@SiO₂ achieved the selective photodegradation of unwanted color constituents present in a dye molecule.

Keywords: Sol-gel process, Metal oxide, Silica matrix, Photodegradation, Photocatalytic activity.

1. Introduction

Zinc oxide (ZnO) has an important semiconducting material because of its typical properties such as resistivity control over the range of 10^{-3} - 10^{-5} Ω cm, band gap of 3.37 eV, large exciton binding energy of 60 MeV at room temperature, high electrochemical stability and non-toxicity. Doping of metal ion into ZnO improves the charge separation in the semiconductor system. [1-3] The azo functional group present in Congo Red (CR) dye, it is commonly found organic pollutant in waste water, in terms of its molecular weight, chemical structure, and azo bonding. CR dye mainly occurs in the effluents discharged from paper, printing, textile and leather industries, and during dyeing operation, about 15% of CR ends up in wastewater. There are many processes to remove CR molecules from coloured effluents and the treatment methods can be divided into three categories: First one is the chemical methods such as ozonation, photo degradation and electrochemical process

and second one is the physical methods such as adsorption and last one is the biodegradation process. [4-6] The effects of metal ion doping on the metal oxide surface, physical structure and photocatalytic activity were investigated very recently [7-10]. However, we focused on the synthesis and photocatalytic activity of ZnO-doped SiO₂ sol-gel matrix. Therefore, in the present work, we report the synthesis of ZnO@SiO₂ matrix. We have studied photodegradation of CR aqueous solution was used as a model pollutant for testing the photocatalytic behavior [11-16]. Thus, photocatalysis is a promising way to eliminate CR and similar organic pollutants from the industrial waste water [17-21].

2. Materials and methods:

2.1. Chemicals

Zinc acetate dihydrate, Tetraethyl orthosilicate and Congo Red were the guaranteed reagents of Sigma Aldrich. Ethanol and nitric acid are of analytical grade and used as received. The aqueous solutions were prepared by using double distilled water. The chemical structure of Congo Red is shown in Figure. 1 [22-26].

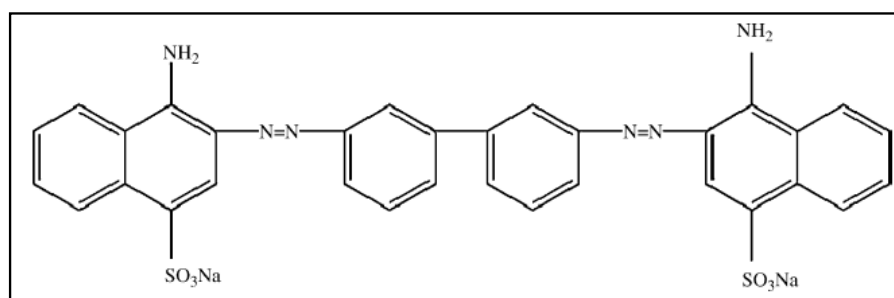


Figure.1 Chemical structure of Congo Red dye

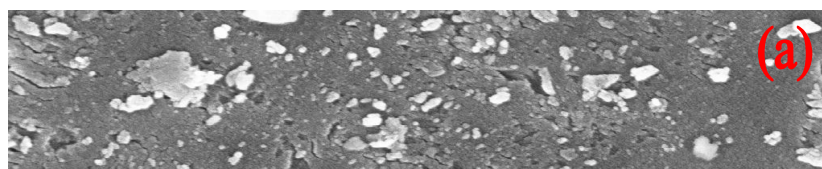
2.2. Synthesis of ZnO doped silica sol-gel matrix

The sol was prepared by using tetraethyl orthosilicate (TEOS), ethanol, double distilled water, Zincacetate dihydrate used as precursor. First, solution A was prepared by mixing TEOS and ethanol in equal volume[27-32]. Then, solution B was prepared by using ZnO precursor and nitric acid together. Nitric acid was added to solution B simply to adjust the pH. Finally, solution B was added to solution A drop wise, stirring vigorously at room temperature. The sol was left to aging, drying and shrinking. The sol-gel substrate was dried at 100°C for 8 h in an hot air oven then calcinated in muffle furnace at 450 °C for 2 hour. The obtained sol-gel material is ground well to form a ZnO@SiO₂ sol-gel matrix[33-37].

3. Results and Discussion

3.1. Surface morphology of metal oxide doped silica material:

SEM microscopy was used for examining the surface morphology and topography of the synthesized materials. It is utilized to characterize the size in diameter and also surface morphology of the synthesized nanoparticles[38-42]. Figure. 2a represent the SEM images of the ZnO@SiO₂ nanoparticles. From the SEM images nanoparticles possess a rough and porous structure, resulting in an large surface area, which provides direct evidence that the ZnO were encapsulated within the silica shell. ZnO@SiO₂ shows particles are almost nano-meter (nm) in range. The solvent polarity was a key factor for the encapsulation processes of the core/shell nanostructures. Elemental analysis of the synthesized nanoparticles was performed using EDX analysis. EDX elemental mapping of the samples are shown in Figure. 2b. EDX analysis confirms Zn, Si and O are present in ZnO@SiO₂[43-45].



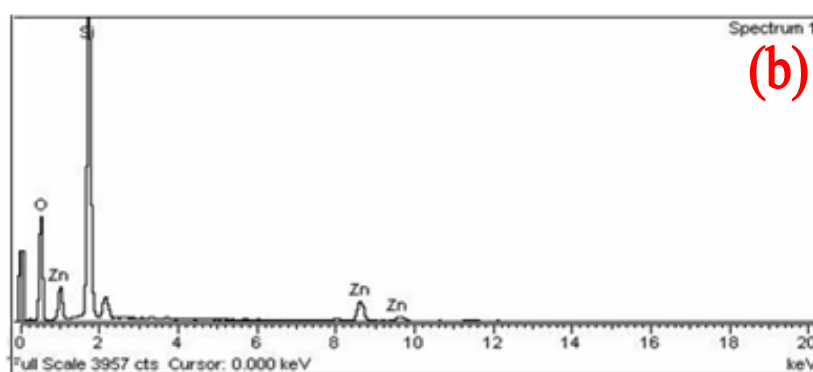


Figure. 2 (a) SEM image of ZnO@SiO₂ nanoparticles and (b) EDX analysis of ZnO@SiO₂.

3.2. FT-IR analysis

FT-IR study on the sol-gel silica structure is shown in Figure. 3 FT-IR spectroscopy of the silica sample showed characteristic absorption band at 954 cm^{-1} and 1030 cm^{-1} band to the Si–O–Si stretching vibration. The 954 cm^{-1} band are assigned to Si–O groups. The vibration at 1635 cm^{-1} is attributed to the H–O–H deformation and indicates the presence of incorporated water in the SiO₂ network. OH groups of water and silanol are detectable at 3437 cm^{-1} . The IR spectrum demonstrates the presence of the vibration bands of Si–O–Si, H–Si–H and OH typical of SiO₂ particles.

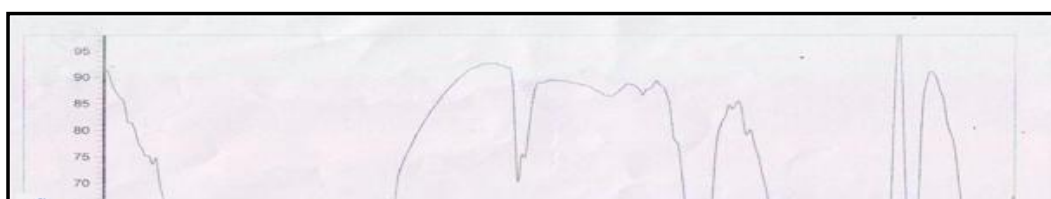


Figure. 3 FT-IR spectrum of silica nanoparticles

3.3. X-ray diffraction analysis

The obtained XRD (Figure. 4) of the metal oxide doped silicate material shown is amorphous in nature. Because of the broad hump, the amorphous silica XRD pattern superimposes Bragg reflexes of quartz. The XRD give much information on the ZnO doping in our study. The diffraction peak at $2\theta = 23.33$ corresponds to (101) plane in SiO_2 and the diffraction peak at $2\theta = 36.08$ and 62.83 corresponds to (101) and (103) planes of the hexagonal ZnO structure, respectively.

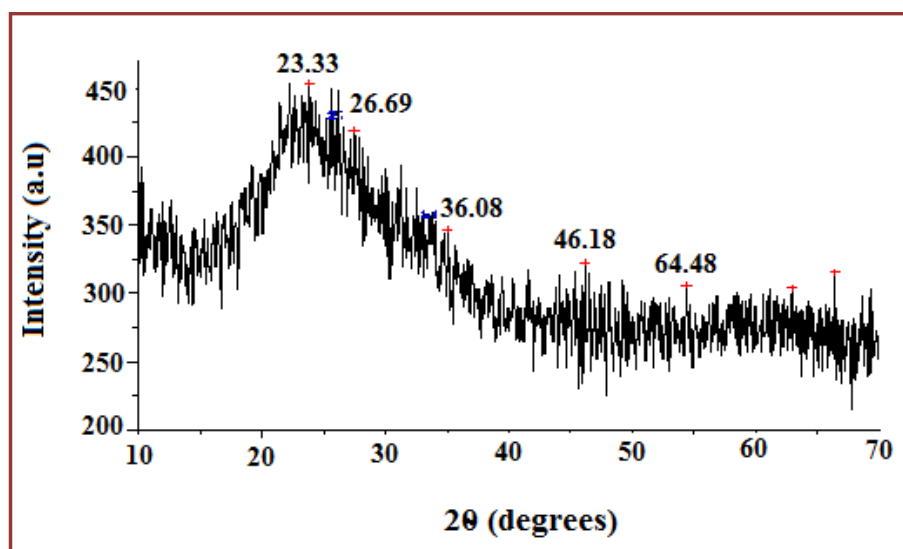


Figure. 3 XRD spectrum of ZnO@SiO₂ nanoparticles

3.4. Photodegradation and decolorization of Congo Red (CR)

We have chosen the photodegradation of CR as model dye pollutant with UV irradiation to evaluate the photocatalytic activity of the proposed photocatalyst like ZnO@SiO₂. The photodegradation was studied using multi lamp photoreactor with mercury UV lamps of 365 nm. The measured wavelength of CR reaction solution is 624 nm. CR of 0.001 mM concentration was prepared and used for this study. The photocatalyst 20 mg was loaded in a reaction chamber. Initially the colour of the dye solution is red, after photodegradation takes place; the color of the solution is changes to colourless (decolourization). The calculated reaction rate and related results are same to be reproducible. The dark reaction carried out in 30 min then the photodegradation were carried out in photoreactor at 20 min interval, corresponding UV-spectrum and plot the time Vs conc. graph was mentioned the degradation of CR. The Time Vs Concentration graph shows the decolorization of CR. The initial dye concentration (0.001 mM) of CR was decreased with increasing irradiation time. (Figure. 4)

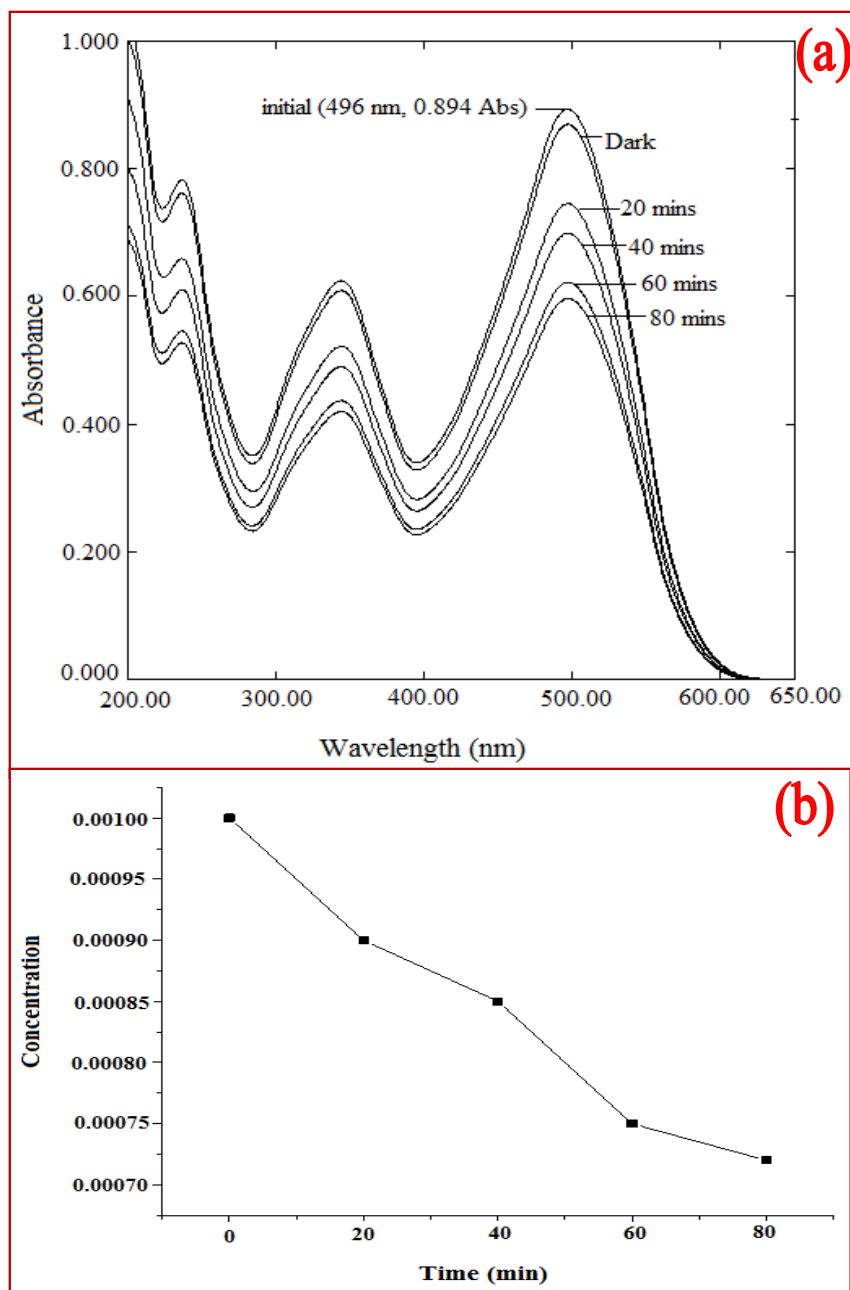


Figure. 4 (a) Absorption spectra changes of CR solution and (b) The time course of decreasing concentration as a function of the photocatalyst

4. Conclusions

ZnO-doped Silica material was synthesized by simple and cost effective sol-gel method. The material was characterized by SEM with EDX, FT-IR and XRD analysis. Photocatalytic activity of the ZnO-doped Silica material was carried out by the photocatalytic degradation of CR as a model pollutant. Thus, photocatalysis is a promising way to eliminate CR pollutants from the aquatic environment.

Conflict of interest

The author declares no competing financial interest.

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