



Sunlight assisted photodecolorization of crystal violet catalyzed by CdS nanoparticles embedded on zeolite A

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ARTICLE INFO

Article history:

Received 29 March 2011

Received in revised form 18 August 2011

Accepted 20 August 2011

Available online 15 September 2011

Keywords:

Crystal violet

CdS nanoparticles

Photodecolorization

Zeolite A

Sunlight

Heterogeneous catalysis

ABSTRACT

Zeolite A was prepared from waste porcelain as a cheap source of Si. Cadmium sulfide nanoparticles were prepared by a precipitation process after ion exchange of Cd^{2+} into the zeolite A. Fourier Transformation Infrared (FT-IR), X-ray Diffraction (XRD) and Scanning Electron Microscopy (SEM) methods were applied for the characterization of samples. The prepared composite was used as a catalyst in the photodecolorization process of crystal violet dye in aqueous solution under sunlight radiation. UV–vis spectrophotometric measurements were performed to determine the extent of decolorization and mineralization. The extent of mineralization was also confirmed by Chemical Oxygen Demand (COD) and Total Organic Carbon (TOC) concentration determination and High Performance Liquid Chromatography (HPLC) methods. Considering the influence of experimental parameters such as catalyst concentration, dye concentration, solution pH, and temperature, the dye photoelimination process was studied. The optimal operation parameters were found as follows: pH 9, 1 g L^{-1} of catalyst loading and 20 ppm of the dye concentration. Zeolite A and naked CdS nanoparticles did not show a remarkable decolorization efficiency. The decolorization process obeyed first-order kinetics.

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1. Introduction

Nowadays, organic dyes are one of the major groups of pollutants found in wastewaters produced from different industries. It is estimated that around 700,000 tons of dyes are produced annually around the world. From this amount about 20% is unloaded as industrial wastes without previous treatment [1]. Synthetic dyes have considerable structural diversity. The chemical classes of dyes utilized more frequently on industrial scale are azo, anthraquinone, sulfur, indigoid, triphenylmethane and phthalocyanine. Crystal violet (CV) is a triphenylmethane dye used as a biological stain or dermatological agent [2,3]. It is also used in a new analytical technique to determine dextran sulfate sodium (DSS) by resonance Rayleigh scattering (RRS). In this method CV is used as a colored ligand to form a new complex that helps to develop a triple wavelength overlapping technique [4]. It has also great effects in controlling fungal growth, and for this reason it has been used to treat tropical diseases for many years [2]. Despite extensive applications of CV, this dye is a mitotic poisoning agent and should be considered as a biohazard substance [5].

A wide range of methods has been developed to remove dyestuffs from wastewaters, such as adsorption on organic or inorganic matrices,

chemical precipitation and flocculation, oxidation by chlorine, H_2O_2 and ozone electrolysis, electrochemical treatment, and ion pair extraction [6]. But based on investigations, around 15% of triphenylmethane dyes cannot be removed from wastewaters due to inefficient chemical and physical processes [2].

In the past decades heterogeneous photocatalysis process, has been introduced as a cost effective alternative for water remediation. Quick oxidation without the formation of polycyclic products is the advantage of this technique compared to the traditional methods for the elimination of many organic pollutants. In this method when a semiconductor is illuminated by a ray of light with a suitable wavelength, it generates electrons and holes which can further produce free-radicals to decompose a large number of organic pollutants such as dyes [7–9]. Since the reactions mostly take place on the surface, the need for a semiconductor supported by a good adsorbent is much felt due to three potential advantages. They include the power to concentrate pollutants near semiconductor particles, the ability for adsorption of generated intermediates and the capability of reusing adsorbents.

Among the different supports, zeolites are considered to be important owing to their special properties such as: unique nanoscale pores, thermal stability, hydrophobic and hydrophilic properties, eco-friendly nature and ion exchange properties which could be used in efficient photocatalytic system designs [10]. Zeolites are crystalline aluminosilicates with cages and channels that can be used as constrained systems for the preparation of semiconductors (CdS), which have a controlled particle size. Zeolites have amphoteric

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properties. The three coordinated aluminum sites and charge compensating cations that exist in the pores of zeolite act as Lewis acid sites. This is while the framework oxygen atoms neighboring Al (Si-O-Al) are considered as Lewis bases [11,12].

Photocatalysis efficiency can also be increased by using semiconductors in nanoparticle size [13]. In photocatalysis process the total number of electrons/holes on the photocatalyst surface indicates catalyst efficiency. In the case of large particles, recombination of the free carriers is more frequent. In this situation, the number of free charges on the surface is reduced and the photocatalytic activity becomes worse. For nanoparticles, the transportation length of free charges from the interface of crystal to the surface is short. This helps electrons/holes travel to the surface of the nanoparticles faster in order to take part in the reaction process [14].

Utilization of solar energy is a very interesting aspect of science. Solar photocatalysis has therefore become a very important area of research in which sunlight is the source of illumination to perform different photocatalytic reactions. Since visible light is the major component of solar radiation, the development of a stable photocatalytic system, which can be affected by visible light, is indispensable.

Among various semiconductors, TiO₂ exhibits extensive applications in photodegradation of many pollutants. However, TiO₂ absorbs a small portion of solar spectrum in UV region. Other major snags in TiO₂ are its high recombination of the photogenerated electron/hole pairs, poor adsorption, low surface area and difficulty of separation from solution, making it unsuitable for direct application in environmental processes [15]. One of the well-known semiconductors used for degradation of chemical pollutants in water is CdS, which shows light absorption in the visible region due to its narrow band-gap of nearly 2.4 eV [16].

CdS photocatalyst is first evoked by relevant light to create electrons. The electrons are then scavenged by molecular oxygen O₂ to yield the superoxide radical anion O₂^{•-} (Eq. (2)) and hydrogen peroxide H₂O₂ (Eq. (3)) in oxygen-equilibrated media. These new formed intermediates can inter react to produce hydroxyl radical •OH (Eq. (4)). It is well known that the •OH radical is a powerful oxidizing agent capable of degrading most pollutants (Eq. (5)) [17]. One of the drawbacks of CdS is that the positive holes produced by photoexcitation lead to the dissolution of the solid lattice or to coating of the photoactive particle with an insulating layer of elemental sulfur (Eq. (6)). To prevent the deleterious photocorrosion of the semiconductor, a sacrificial donor such as sulfide can be added to scavenge the valence band holes leading to produce disulfide ion (Eq. (7)). The product of this oxidation reaction (disulfide or polysulfide), however, has an absorption that overlaps and interferes with the light absorption of CdS. This can be avoided by conducting the experiment in the presence of sulfite [18]. Sulfite further reacts with disulfide ions to regenerate sulfide ion along with thiosulfate ions that are optically transparent (Eq. (8)). Although sulfite is a toxic additive, it is changed to the other non-toxic forms of sulfur via the last reaction [19]:



The aim of this work is to study photodecolorization of crystal violet using a transition metal sulfide (CdS nanoparticles) incorporated in zeolite A. To achieve this, cadmium sulfide nanoparticles were produced inside of the channels and on the surface of zeolite A, by precipitation after the ion exchange process. The obtained compound (nCdS/A) was used as a photocatalyst for decolorization of CV under sunlight radiation. Although Cd is a toxic element, but due to low solubility of CdS and a short contact time in our investigations leaching of Cd is negligible. In addition high efficiency of the catalyst in this period (degradation efficiency of 80% at 20 min) is advantage of the used catalyst.

2. Experimental

2.1. Materials

Crystal violet (methyl violet, C₂₅H₃₀N₃Cl, C.I. 42555) and all other agents were of analytical grade (Merck and Aldrich). Waste porcelain was prepared from Esfahan glass factory (Iran) and grounded by a mill. The grounded particles, with size of 45 μm or mesh 325, were washed with distilled water to use as a source of Si for preparing zeolite A. Table 1 shows the chemical composition of powdered waste porcelain. Doubly distilled-deionized water was used throughout the experiments. The pH of solutions was adjusted either by sodium hydroxide or hydrochloric acid solution.

2.2. Zeolite preparation

Zeolite A was synthesized from waste porcelain at low temperature via a two step alkali conversion [20]. In this method, during the first step, raw material (12.5 g) was added to a 4 M NaOH solution (50 mL) and heated at 80 °C for 12 h. At the end of the heating period, the reaction mixture was filtered; The Si and Al concentrations in the filtrate were analyzed by ICP and atomic absorption, respectively. The

Table 1
Chemical composition of powdered waste porcelain based on XRF analysis.

Compound	Percent
LOI	8.80
SiO ₂	64.41
Al ₂ O ₃	22.57
K ₂ O	1.47
CaO	0.972
Na ₂ O	0.783
Fe ₂ O ₃	0.438
MgO	0.245
P ₂ O ₅	0.076
TiO ₂	0.068
SO ₃	0.048
SrO	0.039
MnO	0.012
ZnO	0.008
CuO	0.007
Total	99.946

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