

Full Length Article

UV/solar light induced photocatalytic degradation of phenols and dyes by Fe(PS-BBP)Cl₃

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ARTICLE INFO

Article history:

Received 16 September 2017

Received in revised form 14 November 2017

Accepted 8 December 2017

Available online 11 December 2017

Keywords:

Fe(PS-BBP)Cl₃

Methyl orange

Catalytic degradation

Solar light

Phenol

ABSTRACT

A polymer supported complex Fe(PS-BBP)Cl₃ [PS = chloromethylated polystyrene divinyl benzene; BBP = 2,6-bis(benzimidazolyl)pyridine] was prepared and characterized by elemental analyses, FT-IR and electronic spectral studies, AAS, magnetic moment measurement, BET surface area measurement and thermal analysis. Its photocatalytic properties were evaluated by studying the degradation of phenolic compounds (phenol, catechol, hydroquinone, *p*-chlorophenol, *p*-nitrophenol, *o*-cresol, *p*-cresol) and dyes (methyl orange, methyl red, rhodamine B) under UV light illumination to CO₂ and H₂O in the presence of H₂O₂. The effects of important parameters such as concentration of Fe(PS-BBP)Cl₃ and substrates (phenol or methyl orange), amount of H₂O₂, and time have been investigated on the rate of degradation of phenol and methyl orange. Complete degradation of phenol and methyl orange occurred at room temperature with Fe(PS-BBP)Cl₃ under UV radiation in 30 and 120 min respectively. Experimental results indicated that the reactivity of Fe(PS-BBP)Cl₃ is more than its unsupported analogue. The intermediates formed during the process were identified using LCMS. Photoelectrochemical measurements (amperometry) carried out for Fe(PS-BBP)Cl₃ revealed the faster migration of photo-induced charge-carriers. The degree of mineralization was also confirmed by chemical oxygen demand (COD) experiments. The results indicated that these could be effectively decolorized and degraded by photocatalytic method, without generation of any hazardous wastes or by-products as evident from the considerable reduction in COD values. The photocatalytic activity of the supported complex in presence of solar light was also investigated.

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1. Introduction

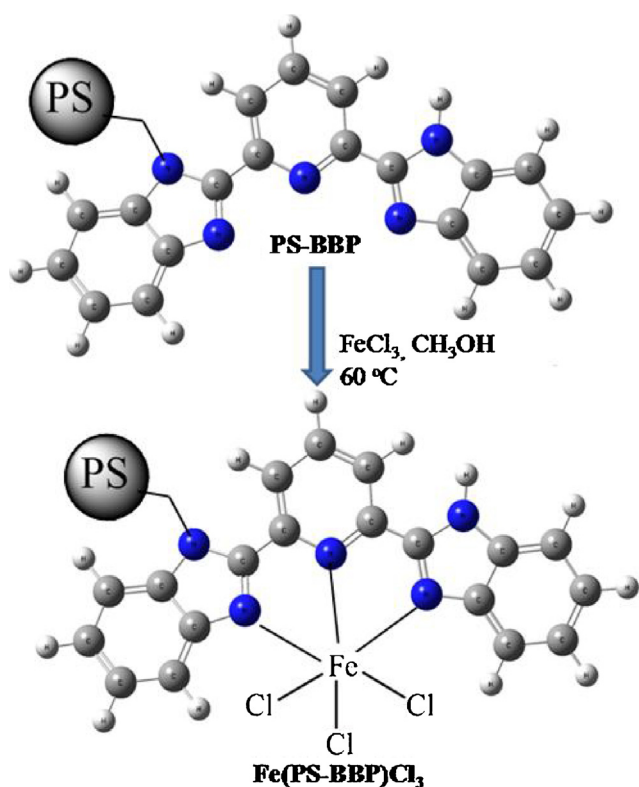
Photocatalysis has emerged as a promising clean advanced oxidation technology, which could address the ever increasing global concerns for environmental pollution abatement based on the utilization of solar and UV energy [1]. Heterogeneous photocatalysis has recently appeared as a new emerging “advanced oxidation process” (AOP), efficient in green chemistry and in production of fine chemicals [2–4]. AOP offer a distinct advantage over many conventional treatment methods, such as biological processes, because faster degradation rates are accomplished and contaminants are degraded rather than transferred from one phase

to another. In addition, there is no requirement for by-product disposal [5]. AOP can be configured using a combination of chemical and physical agents such as a combination of oxidizing agents, an oxidizing agent plus ultraviolet, catalyst or ultrasound and a catalyst plus ultraviolet irradiation [6]. The oxidation using Fenton's reagent, a powerful source of oxidative HO· radicals generated from H₂O₂ in the presence of added Fe²⁺/Fe³⁺ ions [7], has proved to be a promising and attractive treatment method for the effective destruction of a large number of hazardous and organic pollutants [8,9]. The HO· radicals are powerful, non-selective oxidants with the ability of decomposing many organic compounds.

Phenolics pose a serious threat to many ecosystems, water supplies and human health because of their inertness, toxicity, endocrine disrupting abilities and carcinogenic behaviour [10,11]. Despite their low dosage, these pollutants may pose a major health

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Scheme 1. Preparation of Fe(PS-BBP)Cl₃.

hazard due to their mutagenicity and genotoxicity. Phenol and its derivatives have been identified in effluents from petroleum refining, pulp and paper manufacturing, coal processing and have been degraded using photocatalytic oxidation processes in aqueous medium [12–18]. Application of titania catalysts are limited because of its narrow photocatalytic region ($\lambda < 400$ nm) and ability to absorb a small fraction ($< 5\%$) of incident solar irradiation and indoor light, which results from its relatively large band gap (anatase, ~ 3.2 eV). Hence, to improve the photocatalytic efficiency, many studies have been carried out with the aim of eliminating the limitations [19]. TiO₂ nanostructures in poly (methyl methacrylate) (PMMA) composites act as heterogeneous catalysts for photocatalytic removal of dyes, phenols and bacteria from water [20]. Titanium catalyst available in anatase form commercially and ZnO were used for the degradation of phenol [21,22]. Photocatalytic degradation of 2,4,6-trimethylphenol, 2,4,6-trichlorophenol, 2,4,6-tribromophenol, 2,4-dimethylphenol, 2,4-dichlorophenol and 2,4-dibromophenol by TiO₂/UV have been studied [23]. La, Ce, Sm and Nd-doped ZnO nanoparticles as photocatalysts have been successfully fabricated and appeared to be very efficient for the photodegradation of phenols [24–26].

As dyes are used extensively in textiles, foodstuffs, paper and leather industries, these are the most abundant compounds that are discharged into the aquatic environment [27]. Besides, some of the dyes are rather resistant to biodegradation processes under aerobic conditions [28], so their degradation is extremely important to alleviating the damaging impact on the aquatic environment. The photocatalytic decontamination of wastewater containing organic pollutants over the semiconductors has been intensively investigated as an effective economical technique with various advantages of high efficiency, simplicity, good reproducibility, etc., because the photocatalysts can directly utilize sunlight as the energy source [29–34]. Unfortunately, the most widely studied photocatalysts of TiO₂ only absorb the UV light, which

restricts its practical application. To better utilize the visible light of solar energy, a large amount of strategies including metals or non-metal doping, noble metal deposition, dye sensitization, or hetero-junction composite have been adopted to modify TiO₂ to be a visible-light-driven photocatalyst [35,36]. Some metal-organic complexes, such as metal-porphyrin and metal-phthalocyanine, have strong absorption in the visible light region and can activate O₂ or H₂O₂ to oxidize or degrade toxic organic pollutants [37–40], being similar to peroxidases [41,42]. The photocatalytic performance of heterogeneous hydrophobic Cu-Schiff base complex in water treatment of toxic organic pollutants under visible light has been investigated [43].

Compared to the homogeneous Fenton systems, heterogeneous Fenton systems have proven to be promising alternative because of their easy separation. However there are challenges for heterogeneous Fenton systems such as leaching of active metal from the catalyst and lower activities in yielding hydroxyl radicals. Hence, it is desirable to design heterogeneous Fenton like catalyst, which can effectively generate hydroxyl radical with high durability. From the literature survey it was observed that no attention has been paid on the photocatalytic degradation of phenols and dyes using polymer supported complexes. So, in this study, we have synthesized the Fe(PS-BBP)Cl₃ and used it as heterogeneous photocatalyst to degrade phenols and dyes under UV/solar irradiation.

2. Experimental section

2.1. Materials and methods

Chloromethylated poly(styrene-divinylbenzene) spherical beads (PS) with 6% crosslink were obtained as a gift from THERMAX Ltd. India. FeCl₃, *o*-phenylenediamine, 2,6-pyridinedicarboxylic acid, potassium dichromate, 1,10-phenanthroline, ferrous sulphate, mercuric sulphate, ferrous ammonium sulphate, sulphuric acid, potassium hydrogen phthalate, H₂O₂ (30%), phenol, catechol, hydroquinone, *p*-chlorophenol, *p*-nitrophenol, *o*-cresol, *p*-cresol, methyl red, methyl orange, rhodamine B were purchased from Merck (A. R. Grade) and used without further purification. Solvents were freshly distilled by standard methods [44].

The elemental analyses (carbon, hydrogen and nitrogen) of the BBP, PS, PS-BBP and Fe(PS-BBP)Cl₃ were obtained from Elementar Vario Micro Cube CHNS analyzer. Diffuse reflectance spectra were recorded as BaSO₄ disks on a Shimadzu UV-vis-NIR model-3101P spectrophotometer. FT-IR spectra of solid compounds as KBr pellets were taken using a Shimadzu 8400 s FT-IR spectrometer. Thermogravimetric analyses were carried out with a TA instrument, SDT analyzer model Q600. Atomic absorption spectra (AAS) were recorded using an Avanta Pm, GF 3000 instrument. The specific surface area of the Fe(PS-BBP)Cl₃ was measured with a Quanta chrome Nova-1000 surface analyzer using the dynamic Brunner–Emmet–Teller (BET) method in which N₂ gas was adsorbed at 77 K. Far IR spectrum was recorded on a Perkin-Elmer 1700-XFT instrument. Amperometric experiments were carried out using an electrochemical work station (CH Instruments, Texas, USA model: CHI-6194 B) in a standard three-electrode cell configuration. A modified carbon paste electrode of 3 mm diameter was used as a working electrode (1:1 ratio of graphite and Fe(PS-BBP)Cl₃). A Pt wire of 1 mm diameter and Ag/AgCl (saturated KCl) acted as counter and reference electrodes respectively. Residual concentration of phenols and dyes was determined by Shimadzu UV-vis-NIR model-3101P spectrophotometer. LCMS measurements were recorded on a LC-MSD-Trap-XCT-Plus. Magnetic moment of the complex was measured by a VSM model 7404 at IIT Madras.

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