

Photocatalytic reduction of hexavalent chromium in aqueous solution over titania pillared zirconium phosphate and titanium phosphate under solar radiation

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Abstract

The photo-reduction of hexavalent chromium was carried out varying different parameters such as pH, titania loading on zirconium phosphate (ZrP) and titanium phosphate (TiP), initial substrate concentration, irradiation time and catalyst dose. The reduction rate of chromium was favorable under acidic pH than in the alkaline pH. The dissolved oxygen imparts the minimum effect on the reduction of Cr(VI) as oxygen competes with the hexavalent chromium for the electron in the acidic solutions. Hexavalent chromium was completely reduced over titania pillared ZrP and TiP under solar radiation. This process of photo-reduction follows approximately pseudo first-order kinetics.

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1. Introduction

The presence of heavy metals in aquatic bodies has been known to cause pollution problem. The major source of heavy metals is the improper discharge of various industrial wastewaters. These metal ions are generally non-degradable. They have got infinite life times and build up their concentrations in food chains to toxic levels. Among these inorganic pollutants chromium is an important industrial metal that is considered a priority pollutant by US Environmental protection agency. Chromium occurs in two common oxidation states in nature, Cr(III) and Cr(VI), out of which hexavalent chromium is 100 times more toxic than the other (for concentrations higher than 0.05 ppm). This form of chromium is known to be human carcinogen [1] and is generally associated with the development of various chronic health disorders including organ damage, dermatitis and respiratory impairment [2]. In the environment hexavalent chromium salts do not readily precipitate or become bound to components of soil [3]. It is soluble in water and

forms divalent oxyanions: chromate (CrO_4^{2-}) and dichromate ($\text{Cr}_2\text{O}_7^{2-}$). Cr(VI) is mobile in nature and is only weakly sorbed onto inorganic surface. Cr(III) is readily precipitated or sorbed on inorganic and organic substrates.

Cr(VI) is an important contaminant in wastewaters arising from industrial processes such as electroplating, leather tanning or paint making, due to its carcinogenic properties, it has been controlled in many countries. The preferred treatment is to reduce the harmful Cr(VI) to Cr(III) which is less harmful. Again the removal from wastewater is generally accomplished by employing various chemical and physical means such as hydroxide precipitation, ion exchange, adsorption, and membrane process. Recently, a new technology, based on photo-catalysis to eliminate Cr(VI) ions, a toxic pollutant in the environment was applied using solar energy [4,5]. The photocatalytic process was used for rapid and efficient destruction of environmental pollutants.

Reduction by semiconductor photocatalysis technology [6] is a relatively new technique for the removal or recovery of dissolved metal ions in wastewater. Ultraviolet and visible light have sufficient energy to overcome the band gap of semiconductors and form electron-hole pairs (e^-h^+) [7–11]. These charge carriers, which migrate to the semiconductor surface, are

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capable of reducing or oxidising species in solution. The metal is reduced on the semiconductor particle surface. The photocatalytic reduction of chromium using semiconductor particles has been widely studied. This photo-reduction takes place at low pH because the net reaction consumes protons. The photocatalysts used are mainly CdS [12,13], ZnS [12], WO₃ [12,14], various types of TiO₂ [14,15], ZnO [16], titania supported layered compounds (SLCs) [17], titania modified mesoporous silicate MCM-41 [18], sulphated titania [19]. Heterogeneous catalysis and photochemistry principles were used to explain these processes.

The current paper deals with photo-reduction of Cr(VI) using the titania pillared zirconium phosphate and titanium phosphate under solar radiation. The activities of this catalyst was studied under different reaction conditions such as titania load, catalyst dose, substrate concentration, pH, effect of sacrificial electron donors like EDTA, 4-nitrophenol, effect of presence of N₂, O₂, air and were correlated with surface properties.

2. Experimental

2.1. Preparation of the material

Titania pillared ZrP and TiP were prepared from Na-exchanged ZrP and TiP following the procedure reported by Yamanaka et al. [20]. First titania sol was prepared by dropwise addition of titanium(IV) isopropoxide to a vigorously stirred 1 M HCl solution. The resulting slurry was stirred for 3 h to give a clear titania sol and then aqueous suspension of sodium exchanged ZrP or TiP was added to the sol. The suspension was further stirred for 3 h at 50 °C. It was filtered and washed thoroughly with deionised water and dried at different temperatures for further studies.

2.2. Physicochemical characterisation

The X-ray powder diffraction pattern was taken in Philips PW 1710 diffractometer with automatic control. The patterns were run with monochromatic Cu K α radiation, with scan rate of 2° min⁻¹.

UV–vis DRS was taken in Varian UV–vis spectrophotometer in the range of 200–800 nm. The spectra were recorded against boric acid reflectance standard as base line. The computer processing of spectra with CARY 1E software consisted of calculation of Kubelka–munk function $F(R_\infty)$ from the absorbance.

The BET-surface area analysis was carried out using Quantasorb instrument (Quantachrome, USA) by nitrogen adsorption–desorption isotherms at liquid nitrogen temperature (–196 °C). Prior to adsorption–desorption measurements, samples were degassed at 110 °C and 10⁻⁵ Torr for 5 h in vacuum.

FT-IR spectra were taken using Jasco FT-IR-5300 in KBr phase in the range 400–4000 cm⁻¹.

2.3. Experimental procedure

The photo-reduction of Cr(VI) (K₂Cr₂O₇, BDH) was performed in batch process by taking 20 mg/L of the substrate

(fresh solution of K₂Cr₂O₇) and 0.2 g/L of catalyst. The solution was exposed to sunlight in closed pyrex flasks at room temperature with constant stirring. The observations were compared with the blank, which was done in dark. The effect of EDTA, 4-nitrophenol, N₂, O₂ and air were studied keeping all other parameters fixed. After irradiation, the suspension was filtered and analysed for Cr(VI) quantitatively by measuring the absorbance at 348 nm using CARY 1E Spectrophotometer (Varian). The detail method of measurement is found in the literature [21].

3. Results and discussion

3.1. Physicochemical characterisation

The XRD patterns (Fig. 1, the values of crystallite size were pointed out in the figure) show that the crystallite size was found to be less in case of 4 wt.% of titania loaded TiP (104.56 Å) support than that of neat TiP (206.624 Å) calcined at 500 °C and similar case was observed in case of 2 wt.% titania loaded ZrP

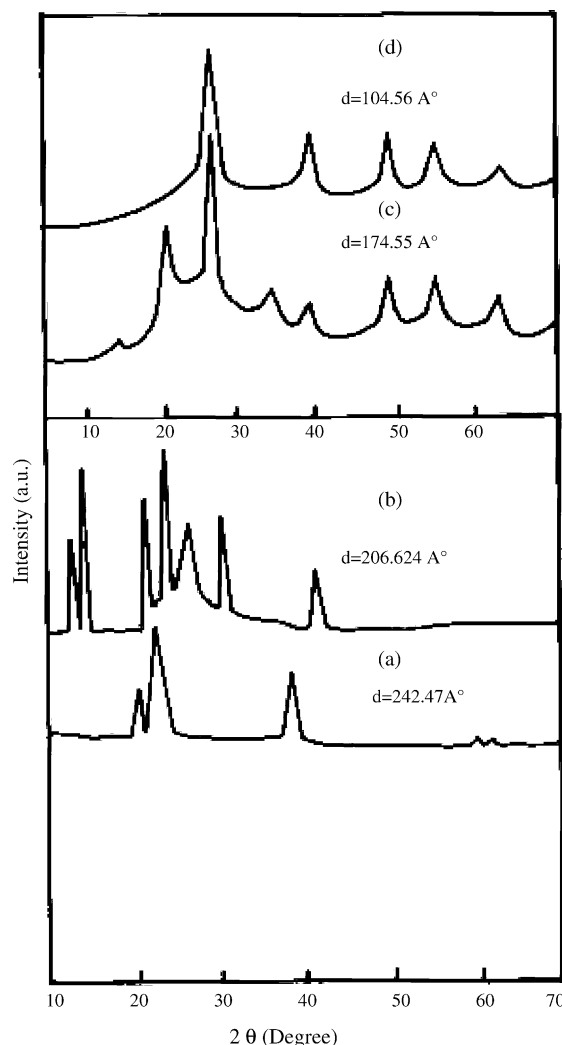


Fig. 1. The XRD pattern of 500 °C calcined (a) ZrP, (b) TiP, (c) ZrP/TiO₂ 2 wt.%, (d) TiP/TiO₂ 4 wt.% (d = crystallite size in Å).

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