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Enhancement of chromium removal efficiency on adsorption and photocatalytic reduction using a bio-catalyst, titania-impregnated chitosan/xylan hybrid film

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ABSTRACT

A novel bio-catalyst, titania-impregnated chitosan/xylan hybrid film was prepared by addition of titanium dioxide in the mixture solution of chitosan and xylan in acetic acid. The structure and surface morphology of the obtained catalyst were characterized by X-ray diffraction, and scanning electron microscope. The adsorption and photocatalytic activity of this photocatalyst were evaluated by photocatalytic reduction of chromium(VI) in aqueous solution under ultraviolet irradiation. From the rough surface modification from xylan addition and the chelating ability of chitosan, the enhancement of chromium(VI) removal by adsorption and photocatalysis of this bio-catalyst was pronounced. The photocatalytic reduction rate of chromium(VI) was 0.56×10^{-3} and 0.03×10^{-3} ppm-min for the titania bio-catalyst and titania nanopowder, respectively. The adsorption of chromium(VI) followed the Langmuir adsorption isotherm model. Pseudo-first order model well described the photocatalytic reduction reactions of the bio-catalyst. Significant dependence of chromium(VI) removal on the titania and chitosan loading can be explained in terms of the relationship between kinetics of chromium(VI) photocatalytic reactions and the loading amount of each chemical. According to the Langmuir–Hinshelwood model, the photocatalytic rate constant of surface reaction of chromium(VI) was increased with an increasing of chemical loading (titania and chitosan) in bio-catalyst. Results from this work exhibited that the novel titania-impregnated chitosan/xylan hybrid film can be a potential material for heavy metal removal in photocatalytic process.

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1. Introduction

Photocatalysis is a promising technology for environmental abatement. As an efficient means for pollution treatment, this technology is widely investigated to remove organic and inorganic contaminants from water and wastewater (Said et al., 2015). Titanium dioxide has been widely used as a photocatalyst due to its activity, photostability, non-toxicity and commercial availability. There are previous studies show that Cr(VI) can undergo photocatalytic reduction and deposit on the surface of titanium dioxide (Kajitvichyanukul and Watcharenwong, 2003). However, TiO_2 exhibits low adsorption activity and difficulty in separating the catalyst from the effluent. The latter problem could be avoided by using the TiO_2 films applied on different types of substrates such as stainless steel (Kajitvichyanukul and Amornchat, 2005) and glass plate (Kajitvichyanukul et al., 2005). As a result, TiO_2 is widely applied in an immobilized form to treat pollutants from water or wastewater. In this recent work, the synthesis of new bio-catalyst as TiO_2-impregnated chitosan/xylan hybrid film was purposed with the primary objective is to enhance the adsorption ability of TiO_2 in photocatalytic process. Chitosan is biopolymer material produced from N-deacetylation of chitin. It has a superb ability in adsorption pollutants especial metal ions (Leceta et al., 2013). It is a powerful chelating agent, which is easy to form complexes with the combination of chitosan on TiO_2 film. It is expected that the adsorption ability of TiO_2 would be enhanced.

In the present paper, a novel bio-catalyst material employing nanoTiO_2 impregnated chitosan/xylan film (TCF) was designed using a simple surface coating method. Both chitosan and xylan are natural nontoxic biomaterials with low cost and easy availability from biomass. Xylans polysaccharides are the group of hemicelluloses that are found in plant cell walls and some algae. They

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contain predominantly β-D-xylose which are linked by β-1,4-glycosidic bonds and branched by α-1,2-glycosidic bonds with 4-O-methylglucosidic acid groups (Dodd et al., 2011). Recently, the hybrid materials from the complexing of chitosan with xylan have shown ability to form selective stable complex with heavy metals (Dai et al., 2012). The increasing of highly porous structure in hydrogel of nanoTiO_2 complexing with chitosan and xylan was introduced by Wu et al. (2014). With the modification of the TiO_2 catalyst on the chitosan/xylan hybrid film, the performance in the photocatalytic reaction could be greatly enhanced. However, there are few reports that focused on application of this bio-catalyst in pollutant removal.

In this work, we investigated the application of TCF bio-catalyst for Cr(VI) removal by both adsorption and photocatalysis. Cr(VI) is a toxic heavy metal widely used in many industries and always found in wastewater stream. The risk and hazard of this heavy metal is discussed widely in the previous work (Eastmond et al., 2008). Cr(VI) was selected to be the tested pollutant in this work. The effects of pH, contact time and initial concentration on the adsorption capacities of Cr(VI) on this material were reported. The equilibrium adsorption isotherms of Cr(VI) onto the bio-catalyst were determined by the Langmuir and Freundlich equations. The photocatalytic activity was evaluated by the decrease of Cr(IV) concentration after irradiation. Effects of TiO_2 and chitosan loadings in TCF materials on photocatalytic activity were investigated. The kinetics of photocatalytic reduction of Cr(VI) were also investigated in this work. The results from this work would be useful for the application of the novel organic–inorganic bio-catalyst concerning the heavy metal removal with enhanced adsorption and photocatalytic ability.

2. Materials and methods

2.1. Chemicals and reagents

Xylan, titanium (IV) oxide nanopowder (Degussa P25, <25 nm particle size, 99.7% trace metals basis), and chitosan flake with a molecular weight of 600,000 g/mol were purchased from Sigma–Aldrich, Singapore. The other chemicals used were of analytical grade obtained from Merck, Thailand. All aqueous solutions were prepared using purified water with a resistance of 18.2 MΩ cm.

2.2. Preparation of nanoTiO_2 impregnated chitosan/xylan hybrid films (TCF)

The procedure for the preparation of nanoTiO_2 impregnated chitosan/xylan hybrid films (TCF) was prepared. First, 1.5% w/w chitosan flake was dissolved in a 50 mL of the CH_3COOH. Then, 0.1% w/w xylan and 0.4% w/w of nanoTiO_2 powder were added to the solution and stirred for 8 h with ultrasonic treatment for 15 min at half hour intervals. Subsequently, another 50 mL of 0.1 M CH_3COOH was added. The slurry was stirred continuously for 24 h to obtain the final transparent viscous solution. After this typical procedure, the amounts of nanoTiO_2 (0.2, 0.4, 0.6, and 0.8% w/w) and chitosan flake (1, 1.5, 2 and 2.5% w/w) were varied to obtain different properties of TCF film.

The pieces of 50 mm × 60 mm × 2 mm glass plates were used as support to immobilize the prepared TCF catalyst. The glass plates were cleaned thoroughly and dried before use. Then, the viscous solution was spread on the glass plate and placed to dry at 100 °C inside an oven for 7 h alternately after deposition. The chitosan–xylan hybrid film (CF) was prepared as a control using the same method without the addition of nanoTiO_2. Both TiO_2, CF, and CTF were used further in adsorption and photocatalysis experimental parts.

2.3. Characterization of nanoTiO_2 impregnated chitosan/xylan hybrid films (TCF)

Surface morphology of the TCF, CF, and TiO_2 powder was studied by scanning electron microscope (SEM), a field emission Hitachi S-4500 SEM operated at 15 kV. This SEM is equipped with energy dispersive X-ray spectroscopy (EDS) to examine the elemental composition in the investigated samples. Small pieces of the prepared photocatalyst were peeled off from the glass plate and stuck on stubs using double-sided tape. Before the samples were analysed, they were sputtered with a layer of gold film to prevent the occurrence of charging effect. Whereas, the phase composition of the prepared photocatalyst was studied using the powder and plate XRD technique. The patterns were recorded on a Shimadzu X-ray Diffractometer (XRD-6000) using Cu Kα radiation. Diffraction patterns were taken over the 2θ range 5–60°.

2.4. Adsorption and photocatalysis experiments

For Cr(VI) removal by adsorption and photocatalysis process, the solution was prepared by dissolving potassium dichromate ($K_2Cr_2O_7$) in distilled water. In the adsorption process, five pieces of TCF were placed in the chromium solution. The Cr(VI) solutions were equilibrated in the dark. The samples were taken during the time interval for Cr(VI) analysis to obtain the effect of pH and adsorption isotherm. The adsorption capacity of each adsorption experiment was calculated as the amount adsorbed Cr(VI) on the surface of TCF per gram of TCF used.

For photocatalytic process, prior to irradiation, the TCF was placed in a solution in 1000 mL of photocatalytic reactor. The solution was illuminated by a 10 W germicide lamp with a nominal wavelength range of 254 nm located at the centre of the reactor. Air was bubbled through the reaction solution to ensure a constant supply of nitrogen gas and to give agitation effect to achieve an equilibrium state of Cr(VI) and TCF. The dark adsorption was conducted until it reached equilibrium and, then, a lamp was turned on to illuminate the TCF for 180 min. The Cr(VI) solution was agitated thoroughly by the magnetic stirrer. The 1.0 mL of the sample after illumination was syringed out for Cr(VI) analysis using a 1.5-diphenylcarbarzide (BDH) colorimetric method (Zhou et al., 1993). The Cr(VI) determination was measured at the absorbance at 540 nm in acid solution with a PerkinElmer Lambda 20 UV–vis spectrometer. The residual Cr(VI) concentration in the aqueous solution was plotted as a function of time. The observed rate constant (k_{obs}) from each experimental condition was calculated. All experiments in the adsorption and photocatalytic reactions were duplicated.

3. Results and discussion

3.1. Material characterization of TCF bio-catalyst

Molecular structures of chitosan and xylan are shown in Fig. 1. Both chemicals are biomaterials that are widely available in nature. Chitosan (Fig. 1a) has an amino group in its structure. Xylan (Fig. 1b) contains predominantly β-D-xylose units. Both chemicals presented their unique characteristics in CF and TCF as detected by XRD pattern in Fig. 2. NanoTiO_2 crystalline (Fig. 2a) was mainly composed of anatase phase (as shown by the main peak of 2θ at 25.3°) and rutile phase (2θ at 27.5°). Normally, chitin–chitosan was outstanding demonstrated peak of 2θ in two positions at 9 and 21° (Sankararamakrishnan et al., 2005). However, the broad peak at approximately 21° also indicates some short-range order in the amorphous polymeric structure of the hemicellulose in xylan (Cara et al., 2013). In our work, two peaks at 9 and 21° in XRD spectra

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