COPPER (II) REMOVAL FROM AQUEOUS SOLUTION USING COBALT-DOPED IRON OXIDE POLYVINYL ALCOHOL-ALGINATE BEADS

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ABSTRACT

Copper (II) contaminated water threatens not only the aquatic lives but also human health due to the ability to accumulate in the food chains. Therefore, effective and economical technique must be developed to solve this issue. Although nanoparticles had been found effective in the removal of heavy metal and dye, theirs instability in aqueous solution had limited theirs practical usage. Therefore, in this research, the magnetic nanoparticles of cobalt-doped maghemite (Co-Fe_2O_3) which were synthesized by coprecipitation method using stable ferric and cobalt salts were immobilized in polyvinyl alcohol (PVA)-alginate matrix (in bead form) for the removal of copper (II). X-ray photoelectron spectroscopy (XPS) was then used to identify the copper (II) removal mechanism under photo condition. The effects of various parameters such as pH, catalyst dosage, light intensity and presence of negative organic compounds were investigated. After 180 min of sunlight irradiation exposure, copper (II) concentration was reduced to 1.080 ppm and 0.625 ppm at pH 4 and 6, respectively. This concentration complied with the World Health Organization (WHO) drinking water standard (less than 2.0 ppm). The optimum pH and catalyst dosage investigated were found to be pH 6 and 8% (v/v), respectively. In addition, organic additives like acetate and ethanol were found to enhance the copper (II) removal. The adsorption-desorption test further proved that Co-Fe_2O_3-PVA alginate bead has good recyclability with a drop of only 2.7% copper (II) removal efficiency after five cycles. Therefore, this bead is an attractive and ecofriendly separation tool to be applied in domestic water purification system under the sustainable light illumination.

ABSTRAK

Pencemaran air oleh kuprum (II) bukan sahaja mengancam kehidupan akuatik tetapi juga kesihatan manusia kerana kemampuan untuk berkumpul dalam rantaian makanan. Oleh itu, teknik yang berkesan dan ekonomi mesti dibangunkan untuk menyelesaikan isu ini. Walaupun nanopartikel didapati berkesan dalam penyingkiran logam dan pewarna, tetapi ketidakstabilan nanopartikel dalam larutan akueus telah menghadkan penggunaan secara praktikal. Oleh itu, dalam kajian ini, nanopartikel bermagnetik kobalt-maghemit (Co-Fe_2O_3) telah disintesis melalui proses sepemendakan dengan menggunakan garam kobalt dan besi. Nanopartikel tersebut seterusnya disekatgerak dalam matrik polivinil alkohol (PVA)-alginat (dalam bentuk manik) untuk mengasingkan kuprum (II). Spektroskopi fotoelektron sinar-X (XPS) kemudian digunakan untuk menyiasat mekanisma penyingkiran kuprum (II). Kesan pelbagai parameter seperti pH, dos pemangkin, keamatan cahaya, dan kehadiran pelbagai anion organik sebatian telah disiasat. Selepas 180 min dalam sinaran matahari, kepekatan kuprum (II) telah berkurang kepada 1.080 ppm dan 0.623 ppm masing-masing pada pH 4 dan pH 6. Kepekatan ini telah mematuhi piawai air minuman organisasi kesihatan sedunia (WHO) (kurang daripada 2.0 ppm). pH dan dos pemangkin optimal ialah masing-masing pada pH 6 dan 8% (v/v). Tambahan pula, organik sebatian seperti acetat dan etanol didapati boleh mempercepatkan penyingkiran kuprum (II). Kajian jerapan-nyahjerapan telah membuktikan bahawa manik Co-Fe_2O_3-PVA alginat boleh dikitarguna sebanyak lima kali dengan penyusutan kecekapan penyingkiran kuprum (II) sebanyak 2.7%. Oleh itu, manik ini boleh dijadikan alat pemisahan yang menarik dan mesra alam untuk digunakan dalam sistem penulenan air domestik dengan menggunakan sinaran cahaya yang mampan.

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LIST OF ABBREVIATIONS

C_0	-	Initial concentration of C_0 (mg/L)
C_e	-	Solution equilibrium concentration(mg/L)
Co-Fe_2O_3	-	Cobalt doped maghemite nanoparticle
Copper (II)	-	Cu(II)
EELS	-	Electron Energy Loss Spectrometer
Fe_2O_3	-	Maghemite nanoparticle
FTIR	-	Fourier Transform Infra-red
k_d	-	Physical decay of active site
K_d	-	Intraparticle iffusion rate constant (mg/g.min)
k_p	-	Photoexication constant
L	-	Thickness of the boundary layer (mg/g)
MagneticNanoparticle	-	MNP
PEG	-	Polyethylene glycol
PVA	-	Plyvinyl alcohol
q_{max}	-	Maximum amount of adsorbed Cu(II) per unit mass adsorbent (mg/g)
R_0	-	Initial photoadsorption rate (mg/L.min)
SEM	-	Scanning electron microscope
TEM	-	Transmission Electron Microscope
TiO_2	-	Titanium Oxide
UV	-	Ultraviolet
XPS	-	X-ray photoelectron spectroscopy
XRD	-	X ray diffraction Spectrometer

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CHAPTER 1

INTRODUCTION

1.1 Background of Study

The removal of heavy metals from waters is important due to their ability to accumulate in living tissues throughout the food chain as non-biodegradable pollutants (Sarı *et al.,* 2007). Among various heavy metals, copper application covers a variety of industry such as architecture, tube, pipe, electrical, automotive, seawater industrial, machined products, fuel gas and etc. (Camarillo *et al.,* 2010). Due to its wide range of application, the effect of Cu(II) contamination to the environment has become more significant than other metals. Copper is an important trace element required by humans for enzyme synthesis, tissue and bone development (Akar *et al.,* 2009; Bilal *et al.,* 2013). However, the divalent copper is toxic and carcinogenic when excessive consumption occurs through ingestion. Moreover, intake of excessively large doses of Cu(II) by man leads to severe mucosal irritation and corrosion, widespread capillary damage, hepatic and renal damage and central nervous system irritation followed by depression (Nadaroglu *et al.,* 2010). Traditional metal ion treatment processes included chemical precipitation, ion exchange, electrolysis, reverse osmosis, adsorption, etc.

Although some of these methods may be more effective than the adsorption process under certain operation conditions, adsorption is still favorable since it is economical, efficient, recyclable, and simpler (Ekmekyapar *et al.,* 2006).

Furthermore, most of those conventional methods involve complicated downstream processes and thereby increase the treatment cost. Therefore, the application of nanomaterial as the novel adsorbent to remove the Cu(II) from the water supply has received much attention recently. In the study of Tizaoui *et al.* (2012), manganese-activated sand was used as the adsorbent filter for the removal of copper. However, it was found that when the copper concentration increased from 3 to 20 mg/L, the filter capacity reduced by about 50% from 0.020 to 0.011 mg Cu/g sand. Salvatore *et al.* (2013) reported that the copper removal from aqueous solutions by sorption onto mixed alginate/pectin gel beads at pH 5.5. Their findings revealed that the sorption process follows a pseudo second-order kinetic model and the sorption capacity increases with the increasing of pectin percentage in the mixed gel system. The removal of copper from aqueous solutions by low cost adsorbent-Kolubara lignite was also studied by Sonja *et al.* (2012) which revealed that lignite was a very efficient adsorbent material, especially with low copper concentration in aqueous solution. The isothermal test from this study also showed that the adsorption data agreed well with Langmuir isotherm model. In most of the mentioned literatures, the removal of Cu(II) is simply recognized as batch adsorption process and adsorption kinetic studies of Cu(II) removal were carried out normally by using Langmuir adsorption model through simple equation fitting.

Recently, it was revealed that the addition of a metal dopant to many of these oxides (Fe_2O_3, TiO_2 Al_2O_3, SiO_2) was able to increase its affinity towards a specific adsorbent when compared to the native oxide material (Warner *et al.,* 2012). By doping the metal oxide nanomaterials with transition metals or metal ions (Mn, Co, Fe, Ni) or non-metal (N, C, B, S) lead to the enlargement of the light absorption range of the photocatalysts and thereby enhancing the harvesting efficiency of sunlight during the photocatalysis treatment process. Moreover, the doped material is believed to decrease the band gap energy of the nanomaterial (semiconductor) and make the excitation of electron from the valence band to the conduction band much easier. This phenomenon could help in the adsorption rate since the excited electrons can be further utilized to attract or adsorb the dissolved Cu(II) cation.

1.2 Problem Statement

Many previous studies have indicated that the heavy metal uptake process using the native oxide (undoped catalyst) (Idris *et al.*, 2012a; Eisazadeha *et al.*, 2013) is spontaneous and greatly affected by the solution pH and light intensity (Kothari *et al.*, 2009; Waseem *et al.*, 2011; Lee *et al.*, 2012). In order to further increase the performance of the native oxide, metal, metal ions or nonmetal dopants are added to improve the photoactivity of the iron oxide through the enlargement of the light absorption density and thereby enhancing the harvesting efficiency of sunlight during the photo treatment process.

In most of the literatures, Cu(II) was removed through simple adsorption process, the enhancement of Cu(II) removal process under photo condition by semiconductor Co-Fe_2O_3 nanoparticles immobilized PVA-alginate bead has yet to be explored. Therefore, in this study, the removal mechanism of Cu(II) by this novel semiconductor photoadsorbent were studied and investigated by surface characterization instrument. Various physico-chemical parameters such as catalyst loading, light intensity were also investigated and reported in this study to optimize the Cu(II) removal. Instead of using adsorption isotherm and kinetic model that was normally used to describe the photoadsorption process, the modified Langmuir-Hinshelwood model and Weber-Morris model were used to describe the Cu(II) photo removal kinetic.

1.3 Objectives

The project is performed with the following objectives:

i) To synthesis and characterize the cobalt doped-maghemite ferrofluid which can be embedded in the PVA-alginate beads.
ii) To synthesis and characterize the cobalt doped-maghemite PVA-alginate beads which can be used to remove Cu(II) from aqueous solution.
iii) To study the effect of parameters such as pH, light intensity, catalyst dosage and present of anionic organic compounds that can influence the Cu(II) removal efficiency of the beads.
iv) To describe the Cu(II) removal by various kinetic models.
v) To study the recyclability of the beads in terms of Cu(II) removal.

1.4 Scope of Research

The scopes of research are as follows:

i) Synthesizing and characterization of the cobalt doped-maghemite ferrofluid by using X-ray diffraction (XRD), transmission electron microscope (TEM), fourier transform infra-red (FTIR) and energy emission loss spectroscopy (EELS).
ii) Synthesizing and characterization of the cobalt doped-maghemite ferrofluid PVA-alginate beads by using fourier transform infra-red (FTIR) and scanning electron microscope (SEM).
iii) The parameters that influence the Cu(II) removal efficiency of the beads such as pH was varied in the range of 4 to 8, catalyst dosage was varied

from 2% (v/v) to 16 % (v/v), light intensity was set in the range of 0-2500 Ft-Cd. Anionic organic compounds (Citrate, acetate, ethanol, ascorbic, hydroquinone) were added so as to evaluate its influences in Cu(II) removal efficiency.

iv) Cu(II) removal was determined by various kinetic models such as Langmuir and Freudlich isotherm, Lagergren kinetic model, Weber-Morris and Langmuir-Hinshelwood equations for the photoadsorbent.

v) The adsorption-desorption experiment was used to investigate the recyclability of the beads for 5 treatment cycles.

1.5 Significant of the Research

This research is aimed to be applied in household heavy metal water treatment to remove Cu(II) which is leaching into the water supply from the chemical industry as effluent through photoadsorption process by using the nanoparticles captured in bead form. In heavy metal contaminated water treatment, iron oxide in maghemite form (γ-Fe_2O_3) is preferred due to its no toxicity, biocompatibility, biodegradability, low particle dimension, large surface area and suitable magnetic properties. The unique form of the maghemite entrapped in the beads result in easy separation of insoluble beads from the water after treatment which do not involved any secondary treatment and thereby reduce the cost of treatment significantly. The beads formed can be reused up to 5 times which was reported by Idris *et al.* (2012a). The performance of each cycle was evaluated and found that the performance was similar as the previous cycle which can be reused up to the five cycles before the beads start to lose its initial property.

Through this research, the photocatalysis performance of the maghemite nanoparticles can be is improved through cobalt doping. Doping of maghemite with transition metals or metal ions is able to increase the photocatalysis efficiency since it can enhance the adsorption of UV light or extend the photoresponse from UV to visible light region. The excitation of electron from valence band to conduction band can also occur easily since the band gap of the semiconductor has been narrowed through doping. This phenomenon can increase the affinity of the photoadsorbent towards the metal cations and remove them from the polluted water by adsorption. Therefore, this kind of photocatalyst has high potential to be applied by the household purification for the treatment of heavy metal-polluted water supply.

1.6 Outline of the Thesis

The thesis is divided into four chapters. The first chapter encompasses the overview of the project which includes objectives, scope and significant of the study. Chapter 2 would surveys latest research on nanoparticles development especially in Cu(II) removal using nanoparticles. In Chapter 3, the methodology and experimentation design of this study were discussed in detail. Chapter 4 discusses the important findings of this research. Finally, Chapter 5 would recapitulates major findings of the thesis, and propose issues inviting future research

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