UNIVERSITI PUTRA MALAYSIA

PREPARATION AND CHARACTERIZATION OF Fe_3O_4/MONTMORILLONITE AND Fe_3O_4/TALC NANOCOMPOSITES FOR REMOVAL OF HEAVY METALS

KATAYOON KALANTARI

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By

KATAYOON KALANTARI

Thesis Submitted to the School of Graduate Studies, Universiti Putra Malaysia, in Fulfillment of the Requirement for the Degree of Doctor of Philosophy

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DEDICATION

To God, who gave me life and strength

To my family. A special feeling of gratitude to my loving parents, Fatemeh Kamalkhani and Abdallah Kalantari, whose words of encouragement and push for tenacity ring in my ears, for all their love, sacrifices and faith and for their bless full prayers.
To my siblings, have never left my side and are very special persons for me.
To my fiancé who stayed with me in difficult time

Abstract of thesis presented to the Senate of Universiti Putra Malaysia in Fulfillment of the requirement for the degree of Doctor of Philosophy

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Chairman: Mansor Ahmad, PhD
Faculty: Science

Toxic metal contamination in water systems is a serious problem threating environment. Many researches have been done to develop effective ways for the heavy metals removal. Traditionally methods used for removal have some disadvantages, such as low efficiency and high cost. Adsorption is one of the techniques use in this area due to its effectiveness and easy operation. In this work, Fe_3O_4/montmorillonite and Fe_3O_4/talc nanocomposites as nanoadsorbent were prepared by co-precipitation method in different percentages. The suitability of the magnetic nanocomposites for the adsorption of Cu^{2+}, Ni^{2+} and Pb^{2+} ions and its efficiency were investigated. The Response Surface Methodology was used for designing the experiment sets. The analysis of variance for Cu^{2+}, Ni^{2+} and Pb^{2+} removal was used to estimate the response of initial concentration of heavy metal ion (mg/L), removal time (s) and dosage of adsorbent (g). Transmission electron microscopy showed that the average nanoparticles size were between 8.24 to 12.88 and 6.62 to 8.13 nm for Fe_3O_4/montmorillonite and Fe_3O_4/talc nanocomposites, respectively. Scanning electron microscopy showed that the nanoparticles were highly uniform in size and spherical shape. X-ray diffraction and Energy-dispersive X-ray, confirmed that magnetic nanoparticles were prepared. Vibrating sample magnetometer reveals the nanoparticles were superparamagnetic. The Fe_3O_4/montmorillonite nanocomposite showed a better capability for adsorption of heavy metals from aqueous solution. The best interpretation for the equilibrium data was given by Langmuir isotherm and the kinetic data showed that the adsorption process followed the pseudo-second order kinetic model for both adsorbents. According to RSM results, for Fe_3O_4/montmorillonite nanocomposite, removal efficiency were 89.72%,94.89%, and 76.15% while the removal time was 120s, Fe_3O_4/montmorillonite nanocomposite amount were 0.06, 0.08 and 0.08 g and initial heavy metal concentrations were 510.16 for Cu^{2+}, 182.94 for Ni^{2+} and 111.90 mg/L for Pb^{2+}. Moreover for Fe_3O_4/talc nanocomposite, removal efficiency were 72.15%, 50.23%, and 91.35% while the removal time was 120s, Fe_3O_4/talc nanocomposite amount was 0.12 g and initial heavy metal concentrations were 100 for Cu^{2+}, 92 for Ni^{2+} and 270 mg/L for Pb^{2+} ions. As a results two unique nano adsorbents made of montmorillonite and talc as a natural

substrate and loaded magnetic nano particles were used to rapid remove of Cu^{2+}, Ni^{2+} and Pb^{2+} ions from aqueous solution. The adsorbents can easily separate by an external magnetic field.

Abstrak tesis yang dikemukakan kepada senate Universiti Putra Malaysia sebagai memenuhi keperluan untuk ijazah Doktor Falsafah

PENYEDIAAN DAN PENCIRIAN FE_3O_4/MONTMORILONIT DAN NANOKOMPOSIT FE_3O_4/TALKUM UNTUK PENYINGKIRAN LOGAM

Oleh

KATAYOON KALANTARI

Januari 2015

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Pencemaran sistem air oleh logam toksik adalah masalah serius yang mengancam alam sekitar. banyak kajian telah dilakukan untuk mencari cara yang berkesan untuk menyingkir logam berat. Kaedah penyingkiran secara tradisi biasanya mempunyai beberapa kelemahan seperti kecekapan yang rendah dan kos yang tinggi. Penjerapan adalah salah satu teknik yang digunakan dalam bidang ini disebabkan keberkesanannya dan operasi yang mudah. Dalam kajian ini, nanokomposit Fe_3O_4/montmorillonite dan Fe_3O_4/talkum sebagai nanopenjerap telah disintesis melalui kaedah pemendakan bersama dalam peratusan yang berbeza. Kesesuaian nanokomposit magnetik untuk penjerapan ion Cu(II), Ni(II) dan Pb(II) dan kecekapannya telah dikaji. Kaedah Respon Permukaan telah digunakan untuk mereka bentuk eksperimen. Analisis varians untuk penyingkiran Cu(II), Ni(II) dan Pb(II) telah digunakan untuk menganggar kepekatan awal ion logam berat (mg/L), masa penyingkiran (s) dan dos bahan penjerap (g). Mikroskopi transmisi elektron dan mikroskopi pengimbasan elektron menunjukkan bahawa purata saiz NPs untuk Fe_3O_4/montmorillonite dan nanokomposit Fe_3O_4/talkum masing-masing adalah di antara 8.24 nm ke 12.88 nm dan 6.62 nm ke 8.13 nm. Pembelauan sinar-X, Tenaga-serakan sinar-X, spektroskopi inframerah Transform Fourier dan magnetometer getaran sampel telah digunakan untuk pencirian. Nanopenjerap magnet menunjukkan keupayaan yang baik untuk menyerap logam berat daripada larutan akueus. Isoterma Langmuir memberikan interpretasi terbaik untuk data keseimbangan dan data kinetik menunjukkan proses penjerapan mengikuti model perintah pseudo-kedua kinetik bagi kedua-dua penjerap. Menurut keputusan RSM, bagi nanokomposit Fe_3O_4/MMT, kecekapan penyingkiran ialah 89.72%, 94.89%, dan 76.15% manakala masa penyingkiran ialah 120 s, kandungan nanokomposit Fe_3O_4/MMT ialah 0.06, 0.08 dan 0.08 g dan kepekatan awal logam berat untuk Cu(II), Ni(II) dan Pb(II), masing-masing ialah 510.16, 182.94 dan 111.90 mg/L.Tambahan pula, untuk nanokomposit Fe_3O_4/talkum, kecekapan penyingkiran ialah 72.15%, 50.23%, dan 91.35% manakala masa penyingkiran ialah 120 s, kandungan nanokomposit Fe_3O_4/talkum ialah 0.12 g dan kepekatan awal logam berat untuk ion Cu(II), Ni(II) dan Pb(II) masing-masing ialah 100, 92 dan 270 mg/L. Hasilnya, dua nanopenjerap unik

diperbuat daripada montmorilonit dan talkum daripada bahan semulajadi dan dimuatkan nanozarah magnet telah digunakan untuk menyingkirkan Cu (II), Ni (II) dan Pb (II) ion dari larutan akueus dengan lebih cepat. Penjerap tersebut boleh dipisahkan oleh medan magnet luar dengan mudah.

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I certify that a Thesis Examination Committee has met on 26th January 2015 to conduct the final examination of Katayoon Kalantari on his thesis entitled '' PREPARATION AND CHARACTERIZATION OF MAGNETIC TALC AND MONTMORILLONITE NANOCOMPOSITES FOR REMOVAL OF HEAVY METALS" in accordance with the Universities and University Colleges Act 1971 and the Constitution of the Universiti Putra Malaysia [P.U.(A) 106] 15 March 1998. The Committee recommends that the student be awarded the Doctor of Philosophy.

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LIST OF ABBREVIATIONS

Fe_3O_4/MMT-NCs	Fe_3O_4/Montmorilonite Nanocomposite
Fe_3O_4/talc-NCs	Fe_3O_4/Talc Nanocomposite
Fe_3O_4 -NPs	Fe_3O_4 nanoparticle
MMT	Montmorillonite
ds	d-spacing
XRD	X-ray Diffraction
TEM	Transmission Electron Microscopy
SEM	Scanning Electron microscopy
EDX	Energy Dispersive X-ray
VSM	Vibrating Sample Management
FTIR	Fourier Transfer Infrared Spectroscopy
AAS	Atomic Adsorption Spectroscopy
BET	Brunauer-Emmett-Teller
CCRD	Central Composite Rotatable Design
RSM	Response Surface Methodology
CCD	Central composite design
ANOVA	Analysis of variance
PRESS	The prediction error sum of squares
CEC	High cation exchange capacity

CHAPTER 1

INTRODUCTION

1.1 Background of Research

The nanotechnology is one of the most widely fields in present study and improvement in fundamental of all technological disciplines, for examples polymer research and wide range of subjects (Paul and Robeson, 2008). As regards nano sized components possess a significant surface area for provided volume (Luo and Daniel, 2003), many essential chemical and physical interactions are influenced through their surface area and surface properties (Hussain et al., 2006).

Recently nanomaterials have got drawn good attention because of they are employed in different applications regarding their outstanding and unique magnetic, optical, catalytic, biological, mechanical, and electrical properties. Application of nanomaterials could improve the environment and control the contamination, which create much more progresses environmental technology and engineering science (Shan *et al.*, 2009).

In nanotechnology field, polymer substrate focused nanocomposites have created a large amount of consideration in recent few years. This field emerged using the cognition that the clays with exfoliated structure can create important mechanical property benefits. Nanocomposites usually are consisting of building blocks components in the size of nm.

The nanocomposite properties are established by the properties of each of the material and by the interactions between various phases and the interfaces between them. Nanocomposites promise to become the trend for the future, getting main ramifications for industries and technological area (Lee and Lichtenhan, 1999).

Recently contamination of water by heavy metal ions has grown as an issue of interest. The activities of human and industries among many sources are the main reason for heavy metals contamination in the aquatic systems (Dirilgen, 2011). As the heavy metal ions are not biodegradable, their existence in water flows and lakes results in bioaccumulation in organisms, that represent a some sort of health hazard to humans Karatas (2012), animals Mishra *et al.* (2012) and, plants (Monferrán *et al.*). Metal ions like Ni(II), Pb(II), and Cu(II) are harmful and carcinogenic at even relatively low level concentration (Liu *et al.*, 2008).

Among these heavy metal ions, Pb(II) is considerable pollution source of water, since they are traditionally used in a number of industrial procedures like metal mining, electronics, and petroleum refining (Freitas *et al.*, 2008).
The industries that use or make the nickel and nickel compounds or mining are the main sources of Ni(II) released to the environment. Contamination with Ni(II) lead to lung cancer (Sen Gupta and Bhattacharyya, 2008).

Copper originates from some industrial activities like printing and electronic processes. Cu(II) is an important element for human body. However, it may cause severe damages in high level concentrations. Many disadvantages have been reported for Cu(II) such as toxicity Chuah *et al.* (2005), neurotoxicity Papandreou *et al.* (2007) and dizzy feeling (Yu *et al.*, 2000).

Numerous techniques have been used for metal ions removing from aqueous solutions (Kul and Koyuncu, 2010). Among these techniques, adsorption method is accepted as one of the most promising and effective approaches (Crini, 2005). The significant benefit of adsorption is that adsorbent has high tendency and great level of loading capacity for heavy metal ion (Li *et al.*, 2010).

Among the available adsorbents, metal nanoparticles are categorized as the best ones for heavy metal ion removal from aqueous solutions. Beside traditional nanoparticles, Fe_3O_4-NPs are gaining increasing attention in remediation of the environment due to the fact that they can be simply separated from water using a magnetic field (Mahdavian and Mirrahimi, 2010).

Additionally, for the recycling and regeneration purpose, Fe_3O_4-NPs based composite adsorbents allowed simple separation from aqueous systems. This kind of facile separating is important to enhance the function performance and reduce the cost during treatment of water and waste water (Zhao *et al.*, 2011).

The modeling and optimization for improving the efficiency of a system are the main stages in an environmental procedure without raising in the cost (Montgomery, 2008). The adsorption processes mechanism is complex. This is to the complicated interaction between parameters and non-linear behavior of these procedures. Consequently, it is very important to determine the optimum experimental condition to get highest efficiency.

The traditional optimization technique (single variable optimization) is not only tiresome and time consuming but also does not illustrate the entire effects of the variables in the procedure and ignores the mixed interaction between physicochemical parameters. This technique can also result in misinterpretation of final results. In order to conquer this problem, a few statistical techniques have been applied. Recently, response surface methodology has drawn great interest since a collection of numerical and statistical methods helpful for examining the effect of several independent parameters (Özdemir and Yapar, 2009).

RSM assesses the interactions between the response (s) and the independent parameters Montgomery (2008) and describes the influence of the independent parameters, alone or combination, in the procedure. This method has many advantages like being more economical, needing fewer experiments number, studding interaction between parameters on response, predicting of the response, checking of method adequacy, and requiring shorter time (Shojaeimehr *et al.*, 2013).

1.2 Problem Statement

Recently Pollution of water and groundwater is one of the most severe problems in environment. Heavy metal ions constitute a significant environmental difficulty because these types of materials usually are not biodegradable and very harmful to

human bodies (Júnior *et al.*, 2009). The metal ions that cause significant problem contain Cu(II), Ni(II), and Pb(II) that are usually related to contaminations and toxic properties.

Many techniques are already designed in order to heavy metal ions pollution removing. Among these techniques, Adsorption is now recognized as an economic and effective technique for heavy metal wastewater treatment. The adsorption process offers flexibility in design and operation and in many cases will produce high-quality treated effluent. In addition, because adsorption is sometimes reversible, adsorbents can be regenerated by suitable desorption process.

Recent research focused on adsorbents such as activated carbons, zeolites, clays, biomass and polymeric materials for the removal of heavy metals. In wastewater treatment, especially in the removal of heavy metals, clays such as montmorillonite have attracted particular attentions, due to their physico-chemical characteristics, low cost, high surface area, availability and the presence of various reactive groups on the surface. Clays present, however some disadvantages that limit their use in practical wastewater treatment applications, such as difficult separation from the liquid phase. Fe_3O_4-NPs on the other hand, have a relatively high surface area, good kinetics for the adsorption and are easy to separate and manipulate in complex multiphase systems with an external magnetic field. Therefore, an excellent, scalable, economic, and non-toxic production of Fe_3O_4-NPs is significantly preferred for potential applications and original study. In addition, the ability and effectiveness of magnetic separation has been showed to be an incredibly technique for solid-liquid phase separation.

However, no material has the properties of both higher saturated magnetization and higher adsorption capacities at the same time. The synthesis of materials with these two properties would be beneficial for their applications in environmental remediation. Fe_3O_4 clay nanocomposites are usually including of Fe_3O_4-NPs to make sure a powerful magnetic response and clay layers to provide favorable functional groups and protection from particle aggregation.
In this study Fe_3O_4/MMT-NCs and Fe_3O_4/talc-NCs were prepared by co-precipitation method for adsorption of metal ions from aqueous solutions optimized by response surface methodology.

1.3 Scope

In this research, using the co-precipitation method, Fe_3O_4-NPs were effectively prepared in the interlayer space of MMT and external surface of talc as solid supports. The Fe ions were reduced to the Fe_3O_4-NPs.

Therefore, the present study represents an attempt toward introducing different percentage of Fe_3O_4-NPs (1, 3, 5, 7, 9 and 12 wt%) into the MMT and talc in. Fe_3O_4/MMT-NCs and Fe_3O_4/talc-NCs were prepared as final products.

The crystalline structure of Fe_3O_4-NPs to all of samples, average size, size distributions, magnetization property, surface morphology and functional groups were characterized using powder X-ray diffraction (PXRD), transmission electron microscopy (TEM), scanning electron microscopy (SEM), energy dispersive x-ray spectroscopy (EDX), vibrating sample magnometer (VSM) and Fourier transform infrared (FT-IR).

The ability of these two nanocomposites were investigated for removal of Cu(II), Ni(II) and Pb(II) ions from solution. Conventional methods of optimization, changing one parameter at a time and keeping the other parameters constant, provide the information related to that particular parameter only. This method of optimization is time consuming and cannot take the mutual interactions of the parameters on the desired outcome. Statistical procedures provide an alternative methodology to optimize a particular process by considering mutual interactions among the variables and give an estimate of the combined effect of these variables on final result. RSM is one such statistical technique and used to optimize the removal efficiency of heavy metals. Three parameters were chosen:

Removal time (s), heavy metal concentration (mg/L) and adsorbent dosage (g).pH of the solution was not changed to avoid of precipitation.

1.4 Objectives

The objectives of the project are:

1. To study the Fe_3O_4/MMT-NCs fabricated by co-precipitation method for removal of heavy metals Pb(II), Ni(II) and Cu(II) through response surface methodology
2. To study the Fe_3O_4/talc-NCs fabricated by co-precipitation method for removal of heavy metals Pb(II), Ni(II) and Cu(II) through response surface methodology
3. To evaluate the adsorption abilities of Fe_3O_4/MMT-NCs as nanoadsorbent for heavy metals Pb(II), Ni(II) and Cu(II) through response surface methodology
4. To evaluate the adsorption abilities of Fe_3O_4/talc-NCs as nanoadsorbent for heavy metals Pb(II), Ni(II) and Cu(II) through response surface methodology

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LIST OF PUBLICATIONS

1. Kalantari, K., Ahmad, MB., Fard Masoumi, H.R., Shameli, K., Basri, M., Khandanlou, R. (2014). Rapid adsorption of heavy metals by magnetic/talc nanocomposite and optimization study using response surface methodology. (*International Journal of Molecular Science,15*, 12913-12927, DOI:10.3390/ijms150712913.

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3. Kalantari, K., Ahmad, MB., Shameli, K., Khandanlou, R. (2014). Size-controlled synthesis of Fe_3O_4 magnetite nanoparticles on the exterior of talc layers. *Research on Chemical Intermediates*, 1-13. DOI: 10.1007/s11164-013-1336-4.

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