UNIVERSITI PUTRA MALAYSIA

PREPARATION, CHARACTERIZATION AND APPLICATION OF ZEOLITE A/METAL OXIDES NANOCOMPOSITES FOR ADSORPTION OF ARSENIC AND LEAD FROM AQUEOUS SOLUTIONS

ABDULLAH ABDULLAH NAJI ALSWAT

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By

ABDULLAH ABDULLAH NAJI ALSWAT

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February 2017

Chairman : Professor Mansor Bin Ahmad, PhD
Faculty : Science

How to completely remove toxic metals from water is still a serious challenge and is the mission that faces researchers and scientists today. In this research, zeolite A/copper oxide (CuO), zeolite A/zinc oxide (ZnO) and zeolite A/iron oxide (Fe3O4) nanocomposites (NCs) were prepared via a co-precipitation method. The effect of weight percentage (wt. %) of these metal oxides (MOs) to the zeolite was investigated to obtain the optimum MOs distribution and surface area. Zeolite-MOs nanocomposites were characterized by Fourier transform infrared (FT-IR) spectroscopy, powder X-ray diffraction (PXRD) and energy dispersive X-ray fluorescence spectrometry (EDXRF). Nitrogen sorption isotherm was used to determine the specific surface areas, pore size and pore volume, while the surface was morphologically observed by transmission electron microscopy (TEM) and field emission scanning electron microscopy (FESEM). The batch adsorption process was used to study the applicability of the nanocomposites to adsorb lead Pb(II) and arsenic As(V) from aqueous solutions. The effects of the adsorbent dosage, pH, initial metals concentration and contact time on the adsorption process were investigated. XRD patterns showed that MOs peaks intensity increased as the intensities of zeolite peaks decreased. TEM images indicated a good distribution of MOs-nanoparticles (NPs) onto the zeolite framework and the cubic structure of the zeolite was maintained. The average particle size of MOs-NPs loaded on the surface of the zeolite was less than 20 nm. The highest surface area was at 8, 5, and 15 wt. % for zeolite A/CuO, zeolite A/ZnO and zeolite A/Fe3O4 NCs, respectively. Results revealed that in 30 minutes, 0.15 g and at pH 5, zeolite A/Fe3O4, zeolite A/ZnO and zeolite A/CuO NCs were able to adsorb 98.2, 93.1 and 83.7% of Pb(II) and 96.8 , 90.1 and 81.3% of As(V), respectively from100 mg/L aqueus solutions. The Langmuir isotherm model showed higher correlation coefficients and provided better agreement with the experimental data, while the adsorption kinetic followed the pseudo-second-order. The sorbents showed an economical and effective way to

adsorb toxic metals due to their ambient operation conditions, low-energy consumption and facile regeneration capability.

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

PENYEDIAAN, PENCIRIAN DAN PENGGUNAAN NANOKOMPOSIT ZEOLIT A/OKSIDA LOGAM UNTUK PENJERAPAN ARSENIK DAN PLUMBUM DARI LARUTAN AKUEUS

Oleh

ABDULLAH ABDULLAH NAJI ALSWAT

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Bagaimana untuk menghapuskan sepenuhnya logam toksik daripada air masih menjadi satu cabaran yang serius dan misi yang dihadapi penyelidik dan ahli sains hari ini. Dalam kajian ini, nanokomposit zeolit A/kuprum oksida (CuO), zeolit A/zink oksida (ZnO) dan zeolit A/ferum oksida (Fe_3O_4) telah disediakan melalui kaedah pemendakan bersama. Kesan peratusan berat (wt.%) logam oksida (MOs) kepada zeolit disiasat untuk mendapatkan taburan MOs dan luas permukaan yang optimum. Nanokomposit zeolit-MOs telah dicirikan dengan Spektroskopi jelmaan Fourier inframerah (FTIR), spektroskopi pembelauan sinar-X serbuk (PXRD), spektrometri sinar-X pendarfluor serakan tenaga (EDXRF). Isoterma serapan nitrogen digunakan untuk menentukan kawasan permukaan tertentu, saiz liang dan liang jilid, sementara permukaan telah morfologi diselidiki dengan menggunakan mikroskopi transmisi elektron (TEM) dan mikroskopi pengimbasan elektron medan pancaran (FESEM). Proses penjerapan kumpulan telah digunakan untuk mengkaji kesesuaian nanokomposit untuk menjerap plumbum (Pb II) dan arsenik (As V) daripada larutan akueus. Kesan dos penjerap, pH, kepekatan mula logam dan tempoh sentuhan pada proses penjerapan telah disiasat. Corak XRD menunjukkan bahawa keamatan puncak MOs meningkat dengan keamatan puncak zeolit berkurangan. Imej TEM menunjukkan taburan oksida logam-nanopartikel (NPs) yang baik ke atas kerangka zeolit dan struktur kubik zeolit dikekalkan. Purata saiz zarah MO-NPs yang dimuatkan di permukaan zeolit adalah kurang daripada 20 nm. Luas permukaan tertinggi ialah pada 8, 5 dan 15 wt.% masing-masing bagi nanokomposit zeolit A/CuO, zeolit A/ZnO and zeolit A/Fe_3O_4. Hasil kajian menunjukkan bahawa dalam masa 30 minit 0.15 g dan pada pH 5, nanokomposit zeolit A/Fe_3O_4, zeolit A/ZnO dan zeolit A/CuO dapat menjerap masing-masing 98.2, 93.1 dan 83.7% bagi Pb II dan 96.8, 90.1 dan 81.3% bagi As V daripada larutan akues 100 mg/L. Model isoterma Langmuir menunjukkan pekali korelasi yang lebih tinggi dan persetujuan yang baik dengan data eksperimen, manakala penjerapan mengikuti kinetik pseudo-tertib kedua. Penjerap menunjukkan cara yang ekonomi dan berkesan untuk

menjerap logam toksik disebabkan oleh keadaan operasi ambien, penggunaan tenaga rendah dan kebolehan permukaan dijana semula.

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I certify that a Thesis Examination Committee has met on 13 February 2017 to conduct the final examination of Abdullah Abdullah Naji Alswat on his thesis entitled "Preparation, Characterization and Application of Zeolite A/Metal Oxides Nanocomposites for Adsorption of Arsenic and Lead from Aqueous Solutions" 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

BET	Brunauere- Emmttee Teller (surface area)
BJH	Barrette Joynere Halenda
C_o	Initial Concentration
C_e	Concentration at equilibrium
EDXRF	Energy dispersive X-ray fluorescence spectrometry
EPA	Environmental Protection Agency
FESEM	Field emission scanning electron microscopy
FT-IR	Fourier transform infrared
FWHM	Full width at half maximum
MOs	Metal Oxides
NCs	Nanocomposites
nm	Nanometer
NPs	Nanoparticles
PXRD	Powder x-ray diffraction
qe	Adsorption capacity
TEM	Transmission electron microscopy
µm	Micrometer
UV-vis	Ultraviolet-visible
VSM	Vibrating sample magnetometer
wt.%	Weight percentage

CHAPTER 1

INTRODUCTION

1.1 Background of the Study

Today, nanotechnology is one of the most popular fields particularly in the current research and in all other technical disciplines. Therefore, nanotechnology consider very promising field in the current century and it is anticipated to rebuild the technological applications in a huge domain of organic, inorganic , semiconductors, biotechnology and energy storage materials.

Nanotechnology defined as; generation, characterization, manipulation and application of materials and devices in the range of less than 100 nm, which could be attributed to the fact that, materials with nanoscale diameter offer distinct properties from those of large materials of the same substance. Further, nanotechnology has the potential to provide; cost effective, efficient and environmentally friendly solutions for the problems that faces global society due to distinguish physical, chemical and biological properties. Further, the nanotechnology commercialization is expected to improve life quality and societal benefits worldwide (Bhattacharyya *et al.* 2009; Pal *et al.* 2015; Sahoo, 2010).

There are several fields of nanotechnology includes; nanostructured materials, nanosystem, nanobiotechnology, nanoelectronics and nanocomposites (Sen *et al.* 1999).

Nanocomposites (NCs) are a significant and promising part of nanotechnology which has attracted great attentions due to their ability to merge the eligible properties of different nanoscales that has several functions and they can be prepared using materials such as, polymers matrices and ceramic strengthened by nanoparticles (Decher, 1997; Lata and Samadder, 2016). NCs topic has now been widely developed, accurately adopted and used worldwide.

The term of 'nanocomposites was first reported by Komarneni, as a composites in which at least one of the phases shows dimensions in the nanometer range (1 nm = 10^{-9} m). Although the term 'nanocomposites' was reported only recently, they are inherent in the biological systems (e.g. bones and plants) (Komarneni, 1992).

On the other hand, metal oxide nanoparticles are classified as promising and desirable sorption materials for toxic metals removal from aqua systems in terms of high selectivity and capacity, consequently, efficient removal of these metals to meet the strict regulations (Hua *et al*., 2012). This is mainly due to their high surface area, high activities and large space for the development of physic and chemical interchanges, reactions, etc.(Uheida *et al*., 2006). Surface modifications of metal oxides nanoparticles with organic and inorganic materials provide hybrid material with higher efficiency and capacity, due to the additional properties that have been introduced by the organic and inorganic attachments and provide more reaction sites especially for toxic metals removal from the environment (Faraji *et al.* 2010).

Zeolites are considered to be promising and important hosts and stabilizers due to their unique features such as: porous structure, high ion exchange capacity, high surface area, hydrophilicity and easily tunable chemical properties, eco-friendly nature and high thermal stability(Xu and Ni, 2010). The nanocomposites can be prepared on two types of zeolites: zeolite-inorganic, and zeolite-organic. Zeolite-inorganic nanocomposites, which include metal or metal oxides supported on zeolite are considered as the most popular, due to the fact that they possess unique properties, molecular selectivity, and hyper functional activity. The use of mesoporous materials such as zeolite, as a support for the metal oxides, has become the focus of intensive research because of the high-performance catalytic activities (Chong *et al*., 2014; Sapawe *et al*., 2013) .

1.2 Problem Statements

Agglomeration of the metal oxides nanoparticles (MONPs) is a major problem that reducing their efficiency and limit their wide applications; decreasing surface area, capacity and selectivity, poor mechanical strength in flow rate system and losing the activity.

Moreover, according to the Environmental Protection Agency, arsenic (As) and lead (Pb) are the most toxic metals in the environment, the ingestion of these metals can cause a number of health problems reach to death.

However, the available techniques and materials for removal of these toxic metals undergo from low adsorption efficiencies and low adsorption capacities that, cannot reach the strict permissible level reported by World Health Organization (WHO) and Environmental Protection Agency (USEPA).

Thus, the development of new nanocomposites with increased capacity , selectivity and affinity to remove these toxic metals and other contaminants is an active emerging part of research in nanocomposites field.

The novelty of this work to overcome the agglomeration problem, we used synthesized zeolite A as stabilizer to prepare three differen kinds of metal oxides nanocomposites. The advantages of using zeolite A, due to its unique properties such; high ion exchange ability, selectivity and its unique super cage pores structure that can control the size metal oxides nanoparticles and their distribution. Moreover, we optimized the weight percentage of metal oxides to get high surface area. That will overcome the agglomeration problem, improve the applicability of MONPs and provide hybrid materials for efficient toxic metals removal.

1.3 Research Approach

In this research, a simple and green co-precipitation method was employed to prepare copper oxide (CuO), zinc oxide (ZnO), and iron oxide (Fe_3O_4) into the cavities and on the surface of the zeolite A framework to produce zeolite/CuO NCs, zeolite/ZnO NCs and zeolite/Fe_3O_4 NCs as hybrid NCs adsorbents to enhance the adsorption efficiency for lead and arsenic adsorption from a water solution without using a capping agent. The weight percentage (wt.%) effect of these metal oxides MOs to the zeolite was investigated to obtain the optimum MOs distribution and highest surface area. Varying wt. % of metal oxides: zeolite provided various morphologies. The prepared NCs were characterized using different techniques; powder X-ray diffraction (PXRD), Fourier transform infrared (FT-IR) spectroscopy, Energy dispersive X-ray fluorescence spectrometry (EDXRF), N_2 adsorption for determining the specific surface areas and pore size, field emission scanning electron microscopy (FE-SEM) and transmission electron microscopy (TEM). The batch adsorption experiment was used to study the applicability of the prepared NCs to adsorb Pb (II) and arsenic As (V) from aqueous solutions. The effects of different adsorption parameters including; pH, adsorbent dosage, initial metals concentration and contact time on the adsorption process were investigated. Atomic absorption spectroscopy (AAS) was used to analyze the samples before and after adsorption process. The adsorption kinetics of lead and arsenic onto prepared NCs were analyzed by two kinetic models; pseudo first-order and pseudo second-order and the Langmuir and Freundlich isotherm models were used to analyzed the obtained adsorption equilibrium data for adsorption of lead and arsenic onto zeolite/MO NCs.

1.4 Objectives

The main objectives of this work are to:

(i) Prepare metal oxides/Zeolite A nanocomposites by incorporating metal oxides nanoparticles of (CuO, ZnO, and Fe_3O_4) using co-precipitation method.
(ii) Determine the optimum conditions to produce nanocomposite with high surface area.
(iii) Determine the optimum conditions for the nanocomposites to remove As(V) and Pb(II) from aqueous solutions.

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