UNIVERSITI PUTRA MALAYSIA

PERFORMANCE OF AMIDOXIME-MODIFIED POLY(ACRYLONITRILE- CO-ACRYLIC ACID) FOR REMOVAL OF BORON IN AQUEOUS SOLUTION

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PERFORMANCE OF AMIDOXIME-MODIFIED POLY(ACRYLONITRILE-*CO*-ACRYLIC ACID) FOR REMOVAL OF BORON IN AQUEOUS SOLUTION

By

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Thesis Submitted to the School of Graduate Studies, Universiti Putra Malaysia, in Fulfilment of the Requirements for the Degree of Master of Science

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PERFORMANCE OF AMIDOXIME-MODIFIED POLY(ACRYLONITRILE-*CO*-ACRYLIC ACID) FOR REMOVAL OF BORON IN AQUEOUS SOLUTION

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Chairman : Professor Luqman Chuah Abdullah, PhD
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Boron is an essential micronutrient for plants, humans and animals, which is also an important component in various industries. Along with the wide spread of boron application, more boron waste pollutes the water sources, and leads to a series of environment and health problems. Adsorption is the most efficient technique among many boron removal technologies; which can treat solutions containing a very low concentration of boron.

This project aimed to produce amidoxime-modified poly(acrylonitrile-*co*-acrylic acid) (AO-modified poly(AN-*co*-AA)) with optimised method and to investigate the performance of AO-modified poly(AN-*co*-AA) on the adsorption of boron ions in batch operation. Batch adsorption was conducted at the physiochemical parameters of pH, adsorbent dosage and initial boron concentration. The isotherms and kinetics of adsorption data were studied at various initial boron concentration. Meanwhile, the Artificial Neural Network (ANN) was simulated from experimental data and applied to optimize, develop and create prediction models for boron adsorption by AO-modified poly(AN-*co*-AA).

As a result, the optimised synthesis method at 55 ºC gave yield of 77% and the conversion of nitrile group to amidoxime at pH = 8 was 78%. The optimal operating condition for boron batch adsorption were determined as initial pH ≈ 7. Besides, the process reached its equilibrium at adsorbent dosage of 4.2 g/L and initial concentration of 41 mg/L. The best fit model for adsorption isotherm was Sips model with heterogeneity factor (n) = 0.7611. In kinetic study, the adsorption data was well fitted in Pseudo-second order equation with equilibrium rate constant (k_2) = 0.0807 ± 0.0112 g. $mg^{-1}.min^{-1}$.

In modelling section, both feed-forward and recurrent artificial neural network (ANN) have been simulated to predict the adsorption potential of synthesised polymer. Among several models, radial basis function (RBF) with orthogonal least square (OLS) algorithm displays good prediction on boron adsorption behaviour with mean square error (MSE) and coefficient of determination (R^2) at 0.000209 and 0.9985, respectively. The adsorption equilibrium is reached within 57 min with the maximum adsorption capacity at 15.23 ± 1.05 mg/g. The results indicate that the AO-modified poly(AN-*co*-AA) is a potential and effective adsorbent for boron removal from aqueous solution.

Abstrak tesis yang dikemukakan kepada Senat Universiti Putra Malaysia sebagai memenuhi keperluan untuk ijazah Master Sains

PRESTASI AMIDOKSIM-DIMODIFIKASI POLI(AKRILONITRIL-KO-AKRILIK ASID) UNTUK PENYINGKIRAN BORON DALAM LARUTAN AKUEUS

Oleh

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Boron adalah mikronutrien yang penting untuk tumbuh-tumbuhan, manusia dan haiwan, di mana ia juga merupakan komponen penting dalam pelbagai jenis industri. Berikutan aplikasi boron yang berleluasa, lebih banyak sisa boron mencemakan sumber air, dan mengakibatkan pelbagai masalah terhadap alam sekitar dan kesihatan. Penjerapan merupakan teknik yang paling efektif antara kesemua teknologi bagi menyingkir boron; di mana boleh merawat larutan yang mengandungi boron pada kepekatan yang rendah.

Projek ini bertujuan untuk menghasilkan amidoksim-dimodifikasi poli(akrilonitril-ko-asid akrilik) (AO-dimodifikasi poli(AN-ko-AA)) melalui kaedah yang telah dioptimasikan dan menyiasat prestasi AO-dimodifikasi poli(akrilonitril-ko-asid akrilik) terhadap penjerapan ion boron dalam operasi kumpulan. Penjerapan kumpulan dijalankan pada parameter fiziokimia pH, dos penjerap dan kepekatan asal ion boron. Data penjerapan isoterma dan kinetik telah dikaji pada kepekatan asal boron yang berlainan. Manakala, *Artificial Neural Network (ANN)* telah disimulasi dari data eksperimen dan diaplikasikan untuk mengoptimasi, membangunkan and membentuk model jangkaan untuk penjerapan boron oleh AO-dimodifikasi poli(AN-ko-AA).

Hasilnya, kaedah sintesis yang baharu memberikan penghasilan 77% pada suhu 55 ºC dan penukaran kumpulan nitril kepada amidoksim ialah 78% pada pH = 8. Model yang paling sesuai untuk isoterma penjerapan ialah model Sips dengan faktor kepelbagaian (n) = 0.7611. Keadaan yang paling optimum bagi penjerapan ialah pH ≈ 7. Proses ini mencapai keseimbangan pada dos penjerapan = 4.2 g/L dan kepekatan asal ion boron = 41 mg/L. Dalam kajian kinetik, data penjerapan paling

sesuai dipadankan dengan persamaan susunan kedua Pseudo dengan kadar tetap keseimbangan (k_2) = 0.0807 ± 0.0112 g. $mg^{-1}.min^{-1}$.

Dalam bahagian pemodelan, kedua-dua rangkaian *feed-forward* dan *recurrent artificial neural* (ANN) telah disimulasi untuk menjangka potensi penjerapan polimer yang telah disintesis. Hasilnya, ANN bertindak sebagai alat jangkaan yang boleh mensimulasi proses penyingkiran. Antara kesemua model, *radial basis function* (RBF) dengan algoritma *orthogonal least square* (OLS) memaparkan jangkaan yang baik terhadap sifat penjerapan boron dengan *mean square error* (MSE) dan *coefficient of determination* (R^2) pada 0.000209 and 0.9985, masing-masing. Keseimbangan penjerapan dicapai dalam 57 min dengan kapasiti penjerapan maksimum pada 15.23 ± 1.05 mg/g. Keputusan menunjukkan bahawa AO-dimodifikasi- poli(akrilonitril-ko-asid akrilik) merupakan penjerap yang berpotensi dan efektif untuk menyingkirkan boron dari larutan akueus.

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Lau Kia Li, June 2019

This thesis was submitted to the Senate of the Universiti Putra Malaysia and has been accepted as fulfilment of the requirement for the degree of Master of Science. The members of the Supervisory Committee were as follows:

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TABLE OF CONTENTS

LIST OF TABLES

LIST OF FIGURES

LIST OF NOTATIONS / SYMBOLS

W_{p0}	dry weight of poly(AN-*co*-AA) (g)
W_c	total weight of copolymer (g)
C_n	conversion percentage of nitrile into amidoxime group (%)
W_{p1}	dry weight of AO-modified poly(AN-*co*-AA) (g)
M_0	total molecular weight of monomers of AN and AA (mol/g)
M_1	molecular weight of amidoxime group (mol.g^{-1})
q_e	adsorption capacity at equilibrium (mg.g^{-1})
C_o	initial concentration of boron ions in solution (mg.L^{-1})
C_e	final concentration of boron ions in solution (mg.L^{-1})
V	volume of boron solution (L)
A_d	amount of adsorbent (g)
$q_{e,cal}$	calculated adsorption capacity at equilibrium (mg.g^{-1})
$q_{e,exp}$	experimental adsorption capacity at equilibrium (mg.g^{-1})
K_L	Langmuir constant(L.g^{-1})
q_m	monolayer saturation capacity (mg.g^{-1})
R_L	Langmuir separation factor
K_F	Freundlich's constant (L.g^{-1})
n_F	Freundlich's exponent (g.L^{-1})
K_S	Sips constant (L.mg^{-1})
n_s	surface heterogeneity
q_t	amount of uptaken ions at given time (mg.g^{-1})
k_1	Lagergren first-order rate constant (min^{-1})
k_2	second order rate constant (g.mg^{-1}.min^{-1})
α	rate of chemisorption
β	surface coverage

k_{id}	intraparticle diffusion rate constant (mg.g^{-1}.min$^{-0.5}$).
f	nonlinear function
y(t)	input of neural network
u(t)	output of neural network
n_u	network input order
n_y	network output order
x_i	ith input
Ø	Gaussian function
w_i	linking weight between the ith node in the hidden and output layer
c	RBF network center
r	distance between RBF network center, c and data points, x
σ	parameter that control the smoothness of RBF
n	number of sampled data
$y_{i,pred}$	ith predicted value by network
$y_{i,exp}$	ith experimental value
$y_{i,avg}$	mean value of $y_{i,exp}$

LIST OF ABBREVIATIONS

AN	acrylonitrile
AA	acrylic acid
ANN	artificial neural network
WHO	World Heatlh Organization
AMF	adsorption membrane filtration
NMDG	N-methyl-D-glucamine
PAN	poly(acrylonitrile)
SSE	sum of square errors
PFO	pseudo-first order
PSO	pseudo-second order
IPD	intra-particle diffusion
FFBPNN	feed-forward back propagation neural network
CFBPNN	cascade-forward back propagation neural network
EFBPNN	elman-forward back propagation neural network
ANN-DE	ANN with differential evolution optimisation
NARX	nonlinear autoregressive model processes with exogenous input
RBF	radial basis function neural network
OLS	orthogonal least squares
SCG	scaled conjugate gradient backpropagation
LM	Levenberg-Marquadt backpropagation
GDM	gradient descent with momentum backpropagation
CGB	conjugate gradient backpropagation with Power Beale restarts
CA	clustering algorithm
BR	Bayesian regularization backpropagation
RP	resilient backpropagation

FTIR	fourier transform infrared spectra
SEM	scanning electron microscopy
CHNS	elemental microanalysis
TGA	thermogravimetric analysis
BET	Brunauer–Emmett–Teller
PZC	point zero charge

CHAPTER 1

INTRODUCTION

1.1 Background

Boron is typically released in the environment from wastewater sources and can be detected mainly in the form of borate salts or boric acid. The main boron sources are urban wastes which originated from a range of various activities and common chemical products casted in agriculture sector (Halim et al., 2013). Boron has negative effects on reproduction and causes impairments of the nervous system. In humans, the symptoms of acute toxicity covers nausea, vomiting, dermatitis, diarrhoea and lethargy (Cengeloglu et al., 2008). Consequently, the exclusion of hazardous boron from wastewater is vital for environment regulation. The European Union has labelled boron as a contaminant of drinking water in national and international drinking water advices (Polat et al., 2004). As recommended by the World Health Organization (WHO), the concentration of boron in the drinking water should be kept below 0.3 mg boron/L (WHO, 2009). There is a legislation condition by Malaysia Department of Environment (DOE) to reduce boron concentration to lower than 1 and 4 mg/L for Effluent Discharge Standards A and B, respectively (Environmental Quality Act, 1974).

Adsorption process can remove or reduce different kinds of pollutants including heavy metals and is widely used in wastewater treatment plant (Bolaños, et al., 2006). Various adsorbents have been attempted to remove various types of heavy metals. The boron adsorption performances have been studied with conventional sorbents such as activated carbon, fly ash and minerals. However, conventional methods are associated with several problems such as low adsorption capacity towards heavy metals. On the other hand, some highly selective technology involves high cost operation (Guan et al., 2016). Therefore, numerous studies have been focused on low-cost and efficient adsorbents materials derived from natural materials for the removal of boron from an effluent (Xu & Jiang, 2008). In this research work, amidoxime-modified poly(acrylonitrile-*co*-acrylic acid) (abbreviated as AO-modified poly(AN-*co*-AA)) which is eco-friendly, reusable and having simpler synthesis method, is used as adsorbent to remove boron ions.

In multivariate modelling, the artificial neural network (ANN) has proven itself to perform an helpful and competent analysis tool in latest years (Bingöl et al., 2012; Mitra et al., 2014; Turan et al., 2011a; Zafar et al., 2017). Artificial Neural Network (ANN) is a reliable method of interpreting data through validation of manmade biological nerves which can compute decisions based on the existing data. ANN handles incomplete information together with more than one input-output situations. ANN provides accurate prediction and does not require exact input-output data relationship. Therefore, ANN has become a preferred tool to study many non-linear or irregular models in engineering applications. It is useful particularly in the condition where the theoretical model is rather problematic to be established while the semi-empirical correlations are weak (Turan et al., 2011b).

There have been many studies on the modelling of heavy metal adsorption by using Artificial Neural Network (ANN). Among them, Turan et. al. (2011a) investigated Zn(II) adsorption from leachate by utilizing biosorbent. Feed forward Backpropagation (FFBPNN) neural network was applied in the study and various BP training algorithms were tried, after which their performances were compared. Besides, Mitra et al. (2014) applied ANN to study the equilibrium of the adsorption of Pb(II) ions by water hyacinth root. However, only a few optimization was conducted using ANN to analyse the adsorption behaviour of boron. One of the research that was carried out was the boron removal using magnetic nanobeads, which applied both Response Surface Methodology (RSM) and ANN in proposing the network (Oladipo & Gazi, 2017). Application of ANN to analyse boron adsorption by using AO-modified poly(AN-*co*-AA) has not been reported elsewhere.

1.2 Problem Statement

Boron is recognised to be important nutrients for all living organisms. The plants require boron to carry out sugar translocation, promote carbohydrate metabolism, facilitate normal growth, hormone action, membrane structure maintenance and nucleic acid synthesis. Damages or retardation on plants bioprocess such as chlorophyll photosynthesis, enzyme reactions, root and leaf growth could happen due to boron deficiency (Guan et al., 2016; Yoshinari & Takano, 2017). For human and animal beings, boron deficiency leads to several health problems such as retarded nervous system function and bone metabolism, low body intake of several minerals and malformations of embryos (Uluisik et al., 2018).

However, there is only small gap between the standard of boron deficiency and excessive boron intake. In the era of industrial revolution, toxicity phenomena due to excessive boron content in water and soil happens at a higher rate than boron deficiency. Boron results in hazardous consequences on living organisms' health depending on both the rate and degree of the exposure (Rioyo et al., 2018). In plants, symptoms of boron toxicity are weaker food synthesis and yield, decline of root cell growth, necrosis and sometimes death of plants (Uluisik et al., 2018). For animals and human, a long-term exposure of boron in the environment should not be overlooked. This can lead to many adverse effects including damages on coronary, cardiovascular, reproductive and nervous (Abdelnour et al., 2018; Nielsen, 2014)

The average boron concentration in brine is approximately 4.6–4.7 mg/L (Tagliabue et al., 2014), whereas its concentration in groundwater has been reported to differ from 0.3 to 100 mg/L (Guan et al., 2016; Hilal et al., 2011; Wang et al., 2014). Many researches on boron adsorption has been done – tempting to remove the metal ions from waste effluent. Membrane-based and thermal desalination technology have been substantially applied in removing boron from irrigation and drinking (Ashfaq et al., 2018; Babiker et al., 2019). Despite its effectiveness in removing boron almost completely from saline water, the efficiency was only around 60% when being applied in domestic water.

Other treatments methods would include ion exchange, adsorption, electrodialysis, membrane bioreactors (MBR) and reverse osmosis (RO) (Babiker et al., 2019). Among them, adsorption and ion exchange technique have been extensively studied. However, ion exchange, electrodialysis, MBR and RO are cost-consuming and do not benefit economically (Bodzek, 2016; Boyang Wang et al., 2014; Wolska & Bryjak, 2013). Besides, A hybrid techniques involving membrane-filtration with ion-exchange has also been conducted for as a mean of boron removal (Babiker et al., 2019). Despite high efficiency in removing the heavy metal, this treatment method was also subjected to high maintenance and operating cost. Instead, adsorption method has become more commonly practiced by industry owners due to its readily usage and desirable cost efficiency (Halim et al., 2013; Kowanga et al. 2016; Recepoğlu et al., 2018). Many adsorbents have been designed for the purpose of boron adsorption, and most of them do contain boron-selective functional groups like N-methyl-D-glucamine (NMDG) (Ide & Hirayama, 2019; Sun et al., 2018), glucose (Sanfeliu et al., 2018) and imino-dipropylene glycol (Yavuz et al., 2013). In addition, some commonly used adsorbent such as activated carbon and fly ash were improved by functional group modification (Guan et al., 2016). However, the mentioned adsorbent are not able to remove boron at very low concentration level. Therefore, more types of adsorbent should be explored, to provide wider choices for industry according to the adsorption capacities, reusability, efficiency in time and other important qualities.

In recent years, the use of amidoxime-functionalized polymer as adsorbent to remove various metal content from wastewater was studied by several researchers (El-Bahy & El-Bahy, 2016; Ji et al., 2016; Yan et al., 2019). The adsorption of metals studied include uranium, cadmium, lead, copper, manganese, and nickel. However, there is lack of research on adsorption of boron by AO-modified poly(AN-*co*-AA).

1.3 Objectives

1. To carry out synthesis and characterisation of AO-modified poly(AN-co-AA)
2. To investigate the kinetic and equilibrium behaviours of boron adsorption process onto AO-modified poly(AN-*co*-AA)
3. To optimise the adsorption process through Artificial Neural Network (ANN).

1.4 Scope of Study

This research proposal is focused on the application of AO-modified poly(AN-*co*-AA) as potential polymer-based adsorbent to isolate boron ions in aqueous solution via batch adsorption. Influence of several operating parameters such as initial boron concentration, pH and adsorbent dosage on the removal efficiency of the developed polymer-based adsorbents were investigated. The equilibrium and kinetics of adsorption process were examined. The adsorption process was optimized with Artificial Neural Network (ANN). The use of different combinations of neural networks and algorithms was explored, considered and finally proposed as effective network choices for boron removal research. At last, the comparison was made between the predicted outputs from multiple neural network models and the experimental data.

1.5 Research Justification

The problem of boron pollution in aqueous solution needs continuous monitoring and surveillance as this element does not degrade and have a tendency to bio-magnify in man through food chain (Nielsen, 2014). The founding of cutting-edge and cost-effective treatment methods is demanded for better cleaning of cationic boron-containing wastewater and recovery of water effluents. The AO-modified poly(AN-*co*-AA) as adsorbent has a wide range of physicochemical properties that enable it as attractive separation media in wastewater treatment and water purification (Gunathilake et al., 2015; Shaaban et al., 2014). It has a large surface area to mass ratio coupled with dual functional groups which gave it the ability to selectively adsorb chemical and biological toxicant to its surface more than ordinary activated carbon (Huang et al., 2013). The successful completion of this research will solve challenges associated with the development of environmentally acceptable functional polymer-based adsorbents for boron-containing effluent treatment.

1.6 Limitations

The adsorption application of AO-modified poly(AN-*co*-AA) was not conducted to boron-containing industrial waste due to time constraint. This shall be an interesting topic since industrial waste has various physical properties and chemical composition that might affect the adsorption behaviours. Besides, basic desorption study was applied in the current work to portray the potential of reusability. However, the reusability study can be conducted in a more detailed way, including variating the pH, time and temperature to observe the effect of physiochemical parameters. These ideas can be served as future project topics.

1.7 Thesis Layout

This thesis consists of 5 chapters and will be organised as follows:

- Chapter 1: Introduction covers brief reviews about the related subject study, problem statement, objectives and scope of the study.
- Chapter 2: Literature reviews containing comprehensive review related to the preparation of amidoxime (AO)-modified resin polymer and boron ions removal from aqueous solution.
- Chapter 3: Material and methods describe the procedure of polymerisation and chemical modifications. The characterization and analysis that have been carried out in this study are described as well.
- Chapter 4: Result and discussions laying out all the observations, lists of data and analysis results with comprehensive discussion
- Chapter 5: Conclusions part that recap all findings that were obtained in this study. The recommendations for future works were included as well.

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LIST OF PUBLICATIONS

Publication(s) that arise from the study are:

Adeyi, A.A., Md Jamil, S. N. A., Abdullah, L. C., Thomas, C.S.T., Abdullah, M., Lau, K.L. (2019) Adsorptive Removal of Methylene Blue from Aquatic Environments Using Thiourea-Modified Poly(Acrylonitrile-co-Acrylic Acid). *Materials*. 12(11): 1374

Adeyi, A.A., Md Jamil, S. N. A., Abdullah, L. C., Thomas, C.S.T., Abdullah, M., Lau, K.L. (2019) Adsorption of malachite green in a fixed bed column by Thiourea modified poly(AN-co-AA). *Chemical Industry & Chemical Engineering Quarterly*, in press.

Adeyi, A.A., Md Jamil, S. N. A., Abdullah, L. C., Thomas, C.S.T., Abdullah, M., Lau, K.L. (2019) Simultaneous adsorption of cationic dyes from binary solutions by thiourea-modified poly(acrylonitrile-co-acrylic acid): Detailed isotherm and kinetic studies. *Materials*. In press.

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