UNIVERSITI PUTRA MALAYSIA

POLYMERIZATION OF THIOUREA MODIFIED POLY(ACRYLONITRILE-CO-ACRYLIC ACID) FOR CATIONIC DYES ADSORPTION FROM SINGLE AND BINARY SOLUTION

ADEYI ABEL ADEKANMI

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By

ADEYI ABEL ADEKANMI

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

August 2019

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DEDICATION

This thesis is dedicated to God, the omnipotent, omniscience and omnipresent.

&

My lovely Adeyi's family

Adeyi, Abel Adekanmi
August 2019

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

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August 2019

Chairman : Professor Luqman Chuah Abdullah, PhD
Faculty : Engineering

Synthetic organic dyes, especially cationic dyes are used extensively as colouring agents in many industries. The discharge of dye-bearing industrial effluents into hydrosphere, generate accumulation of unwanted colours in water, reducing photosynthesis of aqueous flora and causes biological attack towards aquatics. Besides, dye degradation products is also toxic, carcinogenic or mutagenic due to complex dye molecule structure. Hence, proper treatment of dye-containing industrial wastewater is a major environmental pollution issue for consideration. This study investigated the innovation of functional polymer-based adsorbent viz., thiourea modified poly(acrylonitrile-*co*-acrylic acid) that was used to sequestrate selected cationic dyes (malachite green (MG) and methylene blue (MB)) from model effluent by adsorption method. The poly(acrylonitrile-*co*-acrylic acid) copolymer was synthesized by redox polymerization of acrylonitrile (AN) and acrylic acid (AA) monomer, and further modified chemically with thiourea (TU) to produce TU modified poly(AN-*co*-AA) adsorbent. Then, single batch and fixed-bed adsorption experiments for each cationic dye, MG and MB were performed at varied operating conditions. Also, batch and packed-bed mode of adsorption for binary cationic dye solution onto TU modified poly(AN-*co*-AA) was studied.

The adsorption process was found to be pH dependent and the initial dye concentration. The maximum single cationic dye uptake (%) for MG and MB were 92% and 96%, respectively at pH 9. The uptake of both MG and MB was an exothermic process with negative values of ΔH^o and ΔG^o. Equilibrium data were well fitted with Langmuir, Freundlich and Temkin isotherms. The maximum Langmuir adsorption capacity of 605.58 mg/g and 440.81 mg/g was estimated respectively for MG and MB dye. Extended Langmuir model and extended Freundlich model provide a suitable description of the experimental binary data. The comparison

of the single and binary isotherms elucidates an antagonistic interaction between the MG and MB ions. In addition, pseudo-second-order model was found suitable for the description of adsorption kinetic for both dyes onto TU modified poly(AN-*co*-AA), signifying chemisorption between adsorbent and dye molecules.

The single and binary fixed-bed column performance was significantly influenced by pH, concentration of dyes, bed depth and influent flow rate; lower solution pH and higher influent flow rate leads to early breakthrough and exhaustion time, with less adsorption of MG and MB. Conversely, increase in bed-depth resulted in extended breakthrough and saturation time with improved column performance. It was found that Thomas and Yoon-Nelson models perfectly stimulated the adsorption rate and behaviour of cationic dyes entrapment than Bohart-Adams model. Based on experimental findings, TU modified poly(AN-*co*-AA) polymer is a promising functional regenerable adsorbent with high capacity to remove cationic dye (for individual and simultaneous) from liquid environment.

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

PEMPOLIMERAN THIOUREA-DIMODIFIKASI POLI(AKRILONITRIL-KO-AKRILIK ASID) UNTUK PENJERAPAN PEWARNA KATIONIK DALAM SISTEM TUNGGAL DAN BINARI

Oleh

ADEYI ABEL ADEKANMI

Ogos 2019

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Fakulti : Kejuruteraan

Pewarna organik sintetik, terutamanya pewarna kationik telah digunakan secara meluas sebagai agen pewarna dalam pelbagai industri. Pembuangan sisa industri yang mengandungi pewarna ke dalam hidrosfera menghasilkan pengumpulan pewarna yang tidak dikehendaki di dalam air, mengurangkan fotosintesis flora akueus dan mengakibatkan serangan biologi terhadap hidupan akuatik. Selain itu, hasil degradasi pewarna juga adalah toksik, karsinogenik dan mutagenik akibat daripada struktur molekul pewarna yang kompleks. Oleh itu, rawatan yang sewajarnya terhadap sisa air industri yang mengandungi pewarna adalah isu utama pencemaran alam sekitar yang perlu dipertimbangkan. Kajian ini menyiasat inovasi terhadap penjerap berasaskan polimer berkefungsian iaitu thiourea-dimodifikasi poli(akrilonitril-ko-akrilik asid) yang telah digunakan untuk memisahkan pewarna kationik terpilih (malakit hijau (MG) dan metilena biru (MB)) daripada model efluen menggunakan kaedah penjerapan. Poli(akrilonitril-ko-akrilik asid) kopolimer telah disintesis menggunakan pempolimeran redoks terhadap monomer akrilonitril (AN) dan asid akrilik (AA), dan seterusnya dimodifikasi secara kimia dengan thiourea (TU) untuk menghasilkan penjerap TU-dimodifikasi poli(AN-ko-AA). Seterusnya, eksperimen penjerapan kumpulan tunggal dan turus terpadat untuk setiap pewarna kationik; MG dan MB telah dijalankan pada keadaan operasi yang pelbagai. Selain itu, penjerapan mod kumpulan dan turus-terpadat untuk larutan pewarna kationik binari terhadap TU-dimodifikasi poli(AN-ko-AA) telah dikaji.

Proses penjerapan didapati bergantung kepada pH dan kepekatan awal pewarna. Pengambilan pewarna kationik tunggal yang maksimum (%) untuk MG dan MB adalah 92% dan 96%, masing-masing pada pH 9. Pengambilan MG dan MB adalah merupakan proses eksotermik dengan nilai ΔH^o and ΔG^o yang negatif. Data keseimbangan adalah berpadanan dengan isoterma Langmuir, Freundlich dan Temkin.

Kapasiti penjerapan Langmuir yang maksimum untuk MG dan MB dianggarkan sebanyak 605.58 mg/g dan 440.81 mg/g, masing-masing. Model lanjutan Langmuir dan model lanjutan Freundlich menyediakan gambaran yang sesuai untuk data ekperimen binari. Perbandingan antara isoterma tunggal dan binari menunjukkan interaksi anatagonis antara ion-ion MG dan MB. Tambahan lagi, model susunan kedua-pseudo didapati sesuai untuk menggambarkan kinetik penjerapan untuk kedua-dua pewarna terhadap TU-dimodifikasi poli(AN-ko-AA), yang menandakan penjerapan kimia antara penjerap dan molekul pewarna.

Prestasi kolum turus tetap tunggal dan binari dipengaruhi secara signifikan oleh pH, kepekatan pewarna, kedalaman turus, kadar aliran influen; di mana pH larutan yang rendah dan kadar aliran influen yang tinggi menjurus kepada kejayaan dan masa kehausan awal, dengan penjerapan yang kurang terhadap MG dan MB. Sebaliknya, peningkatan kedalaman turus menghasilkan lanjutan kejayaan dan masa ketepuan dengan prestasi kolum yang diperbaiki. Didapati model Thomas dan Yoon-Nelson dirangsang dengan sempurna oleh kadar penjerapan dan sifat pemerangkapan pewarna kationik, berbanding model Bohart-Adams. Berdasarkan dapatan eksperimen, polimer TU-dimodifikasi poli(AN-ko-AA) merupakan penjerap terfungsi yang berpotensi untuk dijana semula dengan kapasiti yang tinggi untuk menyingkirkan pewarna kationik (untuk persendirian dan berterusan) daripada persekitaran cecair.

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Adeyi Abel Adekanmi
August 2019

I certify that a Thesis Examination Committee has met on 28 August 2019 to conduct the final examination of Adeyi Abel Adekanmi on his thesis entitled "Polymerization of Thiourea-Modified Poly(Acrylonitrile-Co-Acrylic Acid) for Cationic Dyes Adsorption from Single and Binary Solution" 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 SYMBOLS

C_0	Initial dye concentrations	mg/L
C_e	Equilibrium dye concentrations	mg/L
C_t	Equilibrium dye concentrations at any time t	mg/L
m	Mass of adsorbent	g
V	Volume of dye solution	L
q_e	Adsorption capacity at equilibrium	mg/g
q_t	Adsorption capacity at any time t	mg/g
$q_{\max}$	Maximum adsorption capacity	mg/g
$q_{e(cal)}$	Calculated adsorption capacity	mg/g
$q_{e(\exp)}$	Experimental adsorption capacity	mg/g
K_L	Adsorption equilibrium Langmuir constant	L/mg
K_{IPD}	Intraparticle diffusion rate constant	mg/g min
K_F	Freundlich constant	$(mg/g)(L/mg)^{1/n}$
R_L	Separation factor	Dimensionless
n	Surface heterogeneity	Dimensionless
C_{IPD}	Boundary layer thickness effect	-
R	universal gas constant	8.314 J/mol K
T	Absolute temperature	K
b_T	Temkin constant related to heat of sorption	J/mol
K_T	Temkin isotherm equilibrium binding constant	L/g
N	number of data points	-
ΔG^o	Change in standard free energy	kJ/mol

ΔH^{o}	Change in enthalpy	kJ/mol
ΔS^{o}	Change in standard entropy	J/mol K
k_1	Pseudo-first-order adsorption rate constant	1/min
k_2	Pseudo-second-order rate constant	mg/g min
α	Elovich sorption rate constant	mg/g min
β	Elovich constant correspond to extent of surface coverage	g/mg
Q	Flow rate	mL/min
Z	Bed height/depth	cm
K_{TH}	Thomas rate constant	mL/(mg.min)
K_{YN}	Yoon Nelson constant	min^{-1}
K_{BA}	Bohart-Adams constant	L/(mg.min)
q_o	Thomas constant for bed capacity	mg/g
q_B	Quantity of dye adsorbed at breakthrough time	mg/g
t_B	Experimental breakthrough time	min
q_{sat}	Quantity of dye adsorbed at bed saturation	mg/g
τ	Time required for 50% adsorbate breakthrough	min
U	Linear velocity	cm/min
N_o	Maximum dye uptake capacity per unit volume of adsorbent column	mg/L
SSE	Sum of squares errors	-
R^2	Correlation coefficient	-

LIST OF ABBREVIATIONS

AN	Acrylonitrile
AA	Acrylic acid
PAN	Poly(acrylonitrile)
Poly(AN-*co*-AA)	Poly(acrylonitrile-*co*-acrylic acid)
TU	Thiourea
KPS	Potassium persulphate
SBS	Sodium bisulphate
MG	Malachite green
MB	Methylene blue
FTIR	Fourier transform infrared
BET	Brunauer-Emmett-Teller
SEM	Scanning electron microscopy
CHNS	Carbon, hydrogen, nitrogen and sulphur
TGA	Thermogravimetric analysis
PFO	Pseudo-first-order
PSO	Pseudo-second-order
MtOH	Methanol
EtOH	Ethanol

CHAPTER 1

INTRODUCTION

1.1 Background of the Study

Water is critical for human life and the survival of almost all ecosystems. There is a great reduction in water availability because the percentage of salt water available is 97.5% compared to the total water volume present on earth (Rehman & Rehman, 2014). According to the United Nations organization reports, there are 1.1 billion people do not have access to a safe supply of drinking water; the majority of them are among the world's most impoverished and developing states (Alfarra et al, 2014). Rapid smash up in population, and industrial activities have ensued in accumulation of biological and chemical contaminants in the environment due to their waste disposal without any treatment (Chaúque et al., 2017; Hokkanen et al., 2016). It is reported that about 1.2 trillion gallons of untreated industrial waste, sewage, and storm water are discharged into the environment annually (Mishra et al., 2017).

Industries such as textile dyeing, metallurgy, battery manufacturing, metal plating, fertilizer production, mining, leather, plastic, rubber, cosmetics, and food are significant contributors that enhance the concentration of contaminant present in wastewater (Albadarin & Mangwandi, 2015; Chen et al., 2017; Song et al., 2016; Yao et al., 2015; Zhang et al., 2016a). Industries, as mentioned earlier, use synthetic dyes which eventually found in the discharged effluents. They are characterized by high molecular weight and complex chemical structures; hence, they are non-biodegradable (Yemendzhiev et al., 2009).

Basic dye ions have become severe threats to human beings and the aquatic ecosystem, due to their toxicity and doggedness after being released into the natural waterbody (Hunger, 2003; Bharathi & Ramesh, 2013; Vandevivere et al., 1998). Synthetic dyes are toxic and lethal; hence they must be removed instantly from aquatic sources, and otherwise, they will lead to a severely detrimental effect on the individual health and on the sustaining diversified flora as well as marine fauna (Elhalil et al., 2016). Thus, the removal of such toxic contaminants from wastewater is a crucial issue globally.

Techniques such as chemical precipitation, membrane filtration, ion exchange, capacitive deionization, low-frequency ultrasonic irradiation, reverse osmosis, water sediment filters (fiber and ceramic), solid block and faucet-mount filters, desalination, electrocoagulation, and adsorption have evolved in the recent past for wastewater treatment (Lacerda et al., 2015; Crini, 2006; Gupta &Suhas, 2009). Precipitation requires high chemical concentration, which is expensive and associated with sludge production. Ion exchange is cost-effective at high concentration, produce secondary pollution from regeneration. Membrane filtration is too costly because it is high energy and membrane restoration cost. Adsorption is the most widely employed technology. However, its application is often restricted to economic factor; the higher the quality, the greater the cost. Selectivity and regeneration of adsorbents are quite expensive, also result in loss of the adsorbency (Kumar & Acharya, 2012).

Adsorbents such as activated carbon, silica (Wawrzkiewicz et al., 2015), natural zeolite (Humelnicu et al., 2017), chitosan (Zhu et al., 2017), graphene oxide (Konicki et al., 2017a) and carbon nanotube/fibre (Zhu et al., 2015), have been produced to isolate the dye from wastewater. In spite of their adsorption capacity, these adsorbents are identified with shortcomings: relatively expensive raw materials, complicated and time-consuming synthesis route, and difficulty in the adsorbent collection. Some adsorbents can adsorb only the specific dye molecule; the goal of selective adsorption is tough to be attained; the adsorption efficiency is relatively low. All these drawbacks seriously hinder the adsorbents applications. Hence, there is a need to search for relatively inexpensive, eco-friendly, multiple functional groups selective material sourced for dye bearing wastewater treatment.

A number of polymer-based adsorbents and their derivatives have been developed, chemically modified and used for the removal of dye ions from aqueous solution, such as polyuria, polythiophenes, polyacrylonitrile (PAN), polydopamine (PDA) microspheres (Fu et al., 2016), amine-tannin-gel (ATG) (Akter et al., 2016), amine-polymer intrinsic microporosity (PIM-1) (Satilmis & Budd, 2017), cellulose nanofibrils aerogel (Jiang et al., 2017), catechol-*co*-polyethylenimine/magnetic nanoparticles (Long et al., 2017), ethylenediamine-modified nano-fibrillated cellulose/chitosan composites (Liu et al., 2016), dithiocarbamate-functionalized graphene oxide (GO-DTC) (Mahmoodi et al., 2017), and poly(levodopa) functionalized MgAl-layered double hydroxide (Zhao et al., 2017).

Recently, many investigations involved functionalization or surface modifications of these polymers in enhancing its effectiveness and selectivity for specific pollutants. For instance, Gupta et al. (2014) synthesized polyaniline zirconium (IV) silicophosphate for the removal of dye from aqueous solution (Gupta et al., 2014). Chen and coworkers prepared poly(cyclotriphosphazene-co-4,4'-sulfonyldiphenol) nanosphere and employed as an adsorbent to the uptake of methylene blue (Chen et al., 2014). Core@shell poly(acrylic acid) microgels/polyethersulfone beads were synthesized by Chen et al. (2017) for the adsorptive removal of dyes (Chen et al., 2017). Surface functionalization of Fe_3O_4 nanoparticles with L-arginine for reactive blue 19 azo dye uptake from the water was also reported by Dalvand and coworkers (Dalvand et al., 2016). Similarly, Zare and team prepared dextrin-g-poly m-phenylenediamine (DgPmPDA) by chemical graft polymerization for the removal of Pb(II) and methylene blue from wastewater (Zare et al., 2018).

However, it is also necessary to investigate the efficacy of these functionalized adsorbents to treat real industrial effluents, both single and binary system studies. In this work, author intend to prepare well-defined polymer based adsorbents, thiourea (TU) modified poly(AN-*co*-AA), understand its structure by characterization and apply the developed polymer-based adsorbents for cationic dyes adsorption from aqueous solution in a single and binary system.

1.2 Problem Statement

Due to swift industrialization and urbanization, the environment suffers high smash up, and a quite large quantity of dangerous and superfluous chemicals are released. Industries such as textile dyeing, battery manufacturing, leather, hair colouring, paper and printing production, cosmetics, food technology, plastic are major contributors enhancing the concentrations of dye components in the environment (Akter et al., 2016). Treatment of industrial wastewater to meet stringent discharge regulations in industrial operations is a major concern globally. Several techniques employed for the removal of cationic dyestuffs from polluted water become ineffective, generate toxic sludge, continuous input of chemicals and highly expensive. The development of economically viable procedures, multi-functional selective sorbents and operating parameters which can isolate toxic, basic dyestuffs from industrial effluents has remained a research focus for several decades.

The most noticeable sign of water pollution is colour. The problem of dyes pollution in water needs continuous monitoring and surveillance as these elements do not degrade and tend to bio-magnify in man through food chain (Mishra et al. 2017). The establishment of cutting-edge and cost-effective treatment approaches is desired for better cleanup of cationic dye-containing wastewater and recovery of water resources. The polymerization of thiourea modified poly(acrylonitrile-co-acrylic acid as adsorbent has a broad range of physicochemical properties that make them particular attractive as separation and reactive media for wastewater treatment and water purification (Lin and Lien 2013; Liu et al., 2018; Zahri et al. 2015). They have 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 multiple times than ordinary activated carbon. The successful completion of this research will solve challenges associated with the development of industrial viable and acceptable functional polymer-based adsorbents for cationic dyes containing effluent treatment.

1.3 Research Goal and Objectives

The main goal of this project is to investigate the efficacy of newly developing functional polymeric adsorbent, TU modified poly(AN-*co*-AA), for adsorptive removal of cationic dyes from aqueous solution. These goals would be achieved via the following objectives:

i. To synthesize poly(acrylonitrile-*co*-acrylic acid) (poly(AN-*co*-AA)) copolymer with different feed mole ratios and chemically modified it with thiourea (TU).
ii. To investigate the uptake of malachite green (MG) and methylene blue (MB) onto TU modified poly(AN-*co*-AA) polymer from aqueous solution in a single dye batch and fixed-bed adsorption systems.
iii. To examine the adsorptive capacity of TU modified poly(AN-*co*-AA) adsorbent for binary cationic dyes (MG and MB) adsorption from aqueous solution in batch and fixed-bed column operations.

1.4 Scope of the Study

This research focused on the preparation and application of TU modified poly(AN-*co*-AA) as a potential large-scale polymer based adsorbents to isolate dyes in industrial wastewater. Influence of several operating parameters such as initial dye concentration, pH, contact time and adsorbent dose on the adsorptive dye removal efficiency of the polymeric adsorbents would be investigated.

Adsorption studies are often limited to batch experiments with single component contaminant (Chengran et al., 2015; Freitas et al., 2017; Janaki et al., 2012), which do not provide adequate scale-up data for possible multicomponent industrial scale wastewater treatment. Knowledge gap exist in adsorption behaviour in dynamic systems, affirming the necessity of this work. Few work have reported the binary dye adsorption using continuous flow conditions, which are more relevant in large scale textile wastewater treatment. Thus, this research also focus on evaluating the binary adsorption of malachite green (MG) and methylene blue (MB) from aqueous solution in a batch and fixed-bed column system. Influence of column operation variables (pH, initial dye concentration, bed depth and flow rate) on binary dye adsorption were examined. The dynamic of the adsorption process were also modeled with the Thomas, Yoon-Nelson, and Bohart-Adams models, to predict the column performance.

1.5 Novelty of Research Study

The functional polymeric adsorbent has a broad range of physicochemical properties that make them particularly attractive as separation and reactive media for wastewater treatment and water purification (Lin & Lien, 2013; Zahri et al., 2015). Industrial wastewaters effluent contain more than one dye component. However, to date, the application of TU modified poly(AN-*co*-AA) polymer in batch and packed-bed adsorption studies to adsorb binary cationic dyes has not been reported elsewhere. Thus, in the present work, evaluation of binary adsorption system was carried out using functional thiourea modified poly(AN-*co*-AA) adsorbent. The mechanism of dyes adsorption onto TU modified poly(AN-*co*-AA) polymer and adsorbent regeneration were investigated in detail, in assessing the industrial viability of the prepared functional adsorbent.

1.6 Thesis layout

This thesis consists of six chapters, organized as:

- Chapter one: Introduction, provides a general introduction on dye bearing wastewater and brief review about the treatment methods, associated problems, objectives, novelty and research scope.
- Chapter two: Presents detail literature review related to modification polymer and its application in dye uptake from the aquatic environment.
- Chapter three: Here, the procedure for polymer synthesis, modification, as well as adsorption experimentation are highlighted.
- Chapter four: Results and discussion concerning polymer yields, characterization, and its application in single system dye adsorption studies.
- Chapter five: Results and discussion of binary cationic dye adsorption studies and analysis are reported here.
- Chapter six: Conclusion and recommendations, this part recap the obtained research findings, limitations and suggestions for feasible future work.

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LIST OF PUBLICATIONS

Adeyi, A. A., Jamil, S. N. A. M., Abdullah, L. C. and Choong, T. S. Y. (2019) Hydrophilic Thiourea-Modified Poly(acrylonitrile-*co*-acrylic acid) Adsorbent: Preparation, characterization and dye removal performance. *Iranian Polymer Journal, 28*(6), 483-491

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