UNIVERSITI PUTRA MALAYSIA

PREPARATION OF NOVEL POLYMERIC ADSORBENT DERIVED FROM MODIFIED RUBBER WASTE FOR THE REMOVAL OF TETRACYCLINE FROM AQUEOUS SOLUTION

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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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Chairman : Associate Professor Abdul Halim Abdullah, PhD
Faculty : Science

The incineration and landfilling of rubber waste generated by the rubber industry contribute to a variety of environmental issues, including land and air pollution. Therefore, this study used rubber waste materials to create cost-effective polymeric adsorbents as unique waste management methods. The rubber waste, collected from dipping tank coagulum (DTC) of a rubber glove manufacturing industry was converted into a promising cost-effective polymeric adsorbent via a simple sulfonation reaction with concentrated sulfuric acid and used to remove tetracycline (TC) from aqueous solutions. The characteristics of rubber waste (RW) and modified rubber waste (MRW) in terms of functional group, point of zero charges, surface area, pore-volume, elemental composition, and morphology were determined. FTIR analysis showed that sulfonate groups ($-SO_3^-$) were successfully added to the surface of RW. The FESEM micrographs revealed that RW and MRW are spherical and irregular, with carbon, oxygen, and sulfur being the dominant components of the rubber waste. RW and MRW are both categorized as mesoporous materials based on their pore size distribution. The pH_{PZC} experiments revealed that RW and MRW have a neutral surface charge at pH 8.78 and 2.62, respectively. The highest TC removal efficiency of 98.78% was achieved in the condition of initial concentration, adsorbent dosage, pH, contact time, and temperature were 30 mg/L, 0.75 g/L, pH 3, 300 minutes, and 25 °C, respectively. The experimental data of TC adsorption were fitted into Langmuir and Freundlich adsorption isotherm models and pseudo-first-order and pseudo-second-order kinetic models. The adsorptions TC onto MRW obeyed the Langmuir isotherm with the maximum capacity of 76.33 mg/g and the pseudo-second-order kinetic models. A possible adsorption mechanism is based on the pH of the solution, pKa value of the TC and pH_{PZC} of MRW. The high adsorption is due to electrostatic interactions and π-π interactions. The thermodynamic data showed that the adsorption of TC on MRW was feasible, spontaneous (ΔG^o = -9.20 to -8.95 kJ/mol), and exothermic

(ΔH^{o} = -4.01 kJ/mol) under the experimental conditions. Based on desorption and reusability studies, the basic medium was the superior eluent for desorbing TC from the surface of MRW. After ten cycles of adsorption-desorption procedure, the percentage removal of TC by MRW remained unchanged at 90%. MRW demonstrated an excellent adsorption capacity for removing TC from real water samples (municipal wastewater and tap water) Based on the results, this study provided a solution to reduce the burden on the rubber industry as well as environmental pollution by converting this rubber waste into a potential low-cost polymeric adsorbent for tetracycline removal from water and wastewater.

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

PENYEDIAAN BAHAN PENJERAP POLIMER NOVEL DARI SISA GETAH DIUBAHSUAI UNTUK PENYINGKIRAN TETRASIKLIN DARIPADA LARUTAN AKUEUS

Oleh

MUHAMMAD ALIYU

November 2021

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Pembakaran dan pelupusan sisa getah yang dihasilkan oleh industri getah menyumbang kepada pelbagai isu alam sekitar, termasuk pencemaran tanah dan udara. Oleh itu, kajian ini menggunakan bahan sisa getah untuk menghasilkan bahan penjerap polimer yang menjimatkan kos sebagai kaedah pengurusan sisa yang unik . Sisa getah (koagulum) yang dikumpulkan dari tangki pencelup dalam sebuah industri pembuatan sarung tangan getah telah ditukar kepada bahan penjerap polimer yang kos efektif melalui tindak balas sulfonasi yang mudah dengan asid sulfurik pekat dan digunakan untuk Mengalih keluar tetrasiklin (TC) dari larutan akueus. Ciri-ciri sisa getah (RW) dan sisa getah diubahsuai (MRW) dari segi kumpulan berfungsi, titik caj sifar, luas permukaan, jumlah liang, komposisi unsur, dan morfologi telah ditentukan. Analisis FTIR menunjukkan bahawa kumpulan sulfonat $(-SO_3^-)$ berjaya ditambah ke permukaan RW. Mikrograf FESEM mendedahkan bahawa RW dan MRW adalah sfera dan tidak teratur, dengan karbon, oksigen, dan sulfur merupakan komponen dominan sisa getah. RW dan MRW kedua-duanya dikategorikan sebagai bahan yang mesoporous berdasarkan taburan saiz liang mereka. Eksperimen pH_{PZC} mendedahkan bahawa RW dan MRW mempunyai caj permukaan yang neutral masing-masing pada pH 8.78 dan 2.62. Kecekapan penyingkiran TC tertinggi sebanyak 98.78% telah dicapai dalam keadaan kepekatan awal, dos bahan penjerap, pH, masa sentuhan, dan suhu masing-masing adalah 30 mg/L, 0.75 g/L, pH 3, 300 minit, dan 25 °C. . Data eksperimen penjerapan TC diuji pada model isoterma penjerapan Langmuir dan Freundlich dan model kinetik tertib pseudo-pertama dan pseudo- kedua. Penjerapan TC pada MRW mematuhi model isoterma Langmuir dengan kapasiti maksimum 76.33 mg/g dan model kinetik tertib pseudo-kedua. Mekanisme penjerapan yang mungkin adalah berdasarkan pH larutan, nilai pKa TC dan pH_{PZC}MRW. Penjerapan yang tinggi adalah disebabkan oleh interaksi elektrostatik dan interaksi π-π. Data termodinamik menunujukkan bahawa penjerapan TC pada

MRW boleh dilaksanakan, spontan (ΔG^{o} = -9.20 hingga -8.95 kJ/mol), dan eksotermik (ΔH^{o} = -4.01 kJ/mol) di bawah keadaan eksperimen. Berdasarkan kajian penyahjerapan dan penggunaan semula, medium berbes adalah eluen terbaik untuk menyahjerap TC dari permukaan MRW. Selepas sepuluh kitaran prosedur penjerapan-penyahjerapan, peratusan penyingkiran TC oleh MRW kekal tidak berubah pada 90% . MRW menunjukkan keupayaan penjerapan yang sangat baik untuk menyingkirkan TC dari sampel air sebenar (air sisa perbandaran dan air paip) Berdasarkan hasilnya, kajian ini memberikan penyelesaian untuk mengurangkan beban industri getah serta pencemaran alam sekitar dengan menukar sisa getah ini menjadi bahan penjerap polimer berkos rendah yang berpotensi untuk penyingkiran tetrasiklin daripada air dan air sisa.

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This thesis was submitted to the Senate of 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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Declaration by Members of Supervisory Committee

This is to confirm that:

- the research conducted and the writing of this thesis was under our supervision;
- supervision responsibilities as stated in the Universiti Putra Malaysia (Graduate Studies) Rules 2003 (Revision 2012-2013) are adhered to.

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%	Percentage
ΔG^o	Free energy of the adsorption
ΔH^o	Enthalpy of the adsorption
ΔS^o	The entropy of the adsorption
<	Less than
>	Greater than
1/T	Reciprocal of temperature
Ce	The concentration of adsorbate after adsorption
Co	The concentration of adsorbate before adsorption
J	Joule
K	Kelvin
K	Rate constant
k_1	The rate constant of the pseudo-first-order kinetics model
K_2	The rate constant of the pseudo-second-order kinetics model
K_F	Freundlich constant relating to adsorption capacity
K_L	Langmuir constant related to the energy of adsorption
L	Liter
M	Molar
mg	Milligram
mg/g	Milligram per gram
mg/L	Milligram per liter
Min	Minute
mL	Milliliter

n	Freundlich isotherm exponent constant
nm	Nanometer
°C	Degree Celsius
pH_{PZC}	Point of zero charge
Qe	Amount of adsorbate adsorbed at equilibrium
Qmax	Maximum adsorption capacity
Qt	Amount of adsorbate adsorbed at time
R	Gas constant
R^2	Correction coefficient
R_L	Separation factor

LIST OF ABBREVIATIONS

AC	Activated carbon
AMX	Amoxicillin
AS	Activated sludge
BET	Brunauer-Emmett-Teller
CBZ	Carbamazepine
CFN	Caffeine
CIP	Ciprofloxacin
CTC	Chlortetracycline
CTP	Citalopram
DFC	Diclofenac
DOC	Doxycycline
DTC	Dipping tank coagulum
EDX	Energy Dispersive X-ray
FESEM	Field Emission Scanning Electron Microscopy
GDTs	Glove dipping tanks
MIPs	Molecularly imprinted polymers
MOFs	Metal-organic frameworks
MRW	Modified rubber waste
MTZ	Metronidazole
NFX	Norfloxacin
NPX	Naproxen
OTC	Oxytetracycline
PC	Paracetamol

PCs	Pharmaceutical compounds
PPs	Pharmaceutical pollutants
RW	Rubber waste
SFN	Sulfonamide
SMX	Sulfachlorpyridazine
TC	Tetracycline
TPM	Trimethoprim
WHO	World health organization
WWE	Wastewater effluent
WWTP	Wastewater treatment plant

CHAPTER 1

INTRODUCTION

1.1 Background of the study

Water quality is now a serious concern that must be addressed to preserve natural ecosystems for the sake of beings and future generations (Ali et al. 2018; Gao et al. 2018; Husein et al., 2019). Pharmaceutical compounds (PCs) are one of the most important groups of pollutants to be concerned about. Previous research indicates that thousands of tons of pharmaceuticals are prescribed and used globally each year, with considerable amounts of them and their metabolites being released into aquatic habitats via manufacturing waste, human or animal faces, hospital or inappropriate disposal runoff (Dong et al., 2019; Gago-Ferrero et al., 2017). The presence of PCs in waterways is a major source of concern because they are almost resistant to biological degradation and retain their chemical structure long enough to cause harm (Tong et al., 2020; Y. Yang et al., 2020). According to Yu et al., (2018), PCs have been detected in some surface water in concentrations ranging from 0.23 to 5.32 µg/L. Water contamination with pharmaceuticals is a worldwide concern due to the negative effects it has on human health and the natural environment. Therefore, municipal and industrial effluent must be treated to a permissible limit threshold before being discharged into bodies of water.

Water separation and purification technologies have received a lot of attention because they play a critical role in regulating the number of pharmaceutical pollutants released from municipal or industrial effluent (Temel et al., 2020). There are various techniques for the removal of pharmaceutical pollutants from wastewater such as advanced oxidation, chemical coagulation, chemical oxidation, and adsorption process (Hasan & Jhung 2015; Torun et al., 2015; Huang et al., 2018). These techniques have several distinct drawbacks. For example, enormous amounts of sludge are created using coagulation processes, and expensive reactants are required when using chemical oxidation methods (González et al., 2016; Napoleão et al., 2016). Among the numerous methods, adsorption is one of the most promising techniques because it is simple, environmentally friendly, fast, and cheap (Jain et al., 2015; Sophia et al., 2018; Temel et al., 2020).

Many studies have focused on the removal of PCs by adsorption. So far, activated carbon (Goel et al., 2004; Ramachandran et al., 2011; Jung et al., 2013; Chang et al., 2015) or zeolites (Lin & Juang, 2009; Kyzas et al., 2015; Dimas Rivera et al., 2021) have been widely used in wastewater treatment as adsorptive materials. Adsorption removal of PCs has considerable promise due to its ease of implementation into existing water treatment systems. Alternatively, adsorbent stability and regeneration drive research and development of new and effective adsorbents derived from polymeric materials. Adsorptive materials such as activated carbon-based adsorption have a high capital cost as well as ineffectiveness and non-selectivity against vat and dispersion pharmaceuticals. Additionally, saturated carbon regeneration is difficult and results in an adsorbent loss (Chang et al., 2015). Adsorbents are either disposed of or regenerated for future usage, depending on demand, cost, and the type of the pollutant to be adsorbed (Chan et al., 2017). Adsorbent regeneration must be simple and environmentally beneficial, restoring valuable adsorbates while minimizing the requirement for virgin adsorbents. Polymeric adsorbents with potential applications in water treatment as a replacement for activated carbon due to their mechanical strength and large surface area (Davidescu et al., 2019). Polymeric adsorbents have high mechanical strength, consistent pore size distribution, and the capacity to regenerate efficiently in a variety of circumstances. Polymeric adsorbents are fouling resistant and have a longevity of up to 2,000 regeneration cycles (T. Yu et al., 2018). Unlike activated carbon, which must be renewed regularly.

1.2 Problem statement and Justification of the study

Pharmaceutical compounds as an emerging pollutant, have generated different challenges and risk implications for water quality. TC is an example of a PCs that are used to treat a variety of microbial infections, including anthrax, chlamydial infections, cholera, Lyme disease, and syphilis (Xu et al., 2020). It's also used to treat farm animals and domestic pets as veterinary medicine (Z. Ma et al., 2020). Because of its effectiveness and low cost, TC has become one of the most widely used antibiotics in the world. Statistics show that nearly 6000 tons of TC are consumed annually in the United States and Europe, with global sales of $ 1.9 billion in 2014 (Z. hui Yang et al., 2019; S. Zhang et al., 2018). Human and animal excretion of the substance in the form of urine, and faeces, as well as improper disposal of expired medicines and leftovers from the drugs, all contribute to TC entry into surface water (Z. Ma et al., 2020). The presence and passage of TC in waterways is a major source of concern. Because TC contributes to increased microbial resistance to similar antibiotics, removing mutant infection strains becomes difficult (Diab et al., 2018; J. Ma et al., 2019). According to Sreejith et al., (2020), TC has been detected in some surface water in concentrations ranging from 0.23 to 5.32 µg/L. Therefore, the existence of traces of TC in the environment can result in the emergence of antibiotic-resistant microorganisms to which humans may be exposed through drinking water (L. Wang et al., 2020). Therefore, an effective technological solution is required to address this issue.

For the removal of TC, several treatment methods such as membrane filtration (Matsubara et al., 2020), advanced oxidation process (Vimonses, 2011; Rioja et al., 2014), or adsorption can be used. Patel et al., (2019) outline the benefits and drawbacks of each of them. In general, the main drawbacks of these methods are their high operational/energy costs, difficulties in implementing on a wide scale, or low removal rates. Adsorption technology solves most of these problems because it can achieve a high percentage removal, requires little energy and mild operating conditions, and there are no by-products introduced to the system (Ali et al., 2018). However, various adsorption issues must be addressed, most notably regeneration and sludge management, as well as the selection of an efficient adsorbent.

The rubber industry is currently struggling with the challenge of regulating massive volumes of rubber waste generated during the rubber glove manufacturing process (Mahapatra et al., 2021). Furthermore, rubber glove manufacturing plants generate large amounts of formulated rubber latex waste known as dipping tank coagulum (DTC), which is primarily produced by rubber glove dipping tanks and It is classified as scheduled waste, which must be disposed of through incineration in accordance to the Department of Environment's Scheduled Waste Regulations (Devaraj et al., 2017). According to Clark (2013), approximately 36000 tons of rubber waste are generated annually from the glove manufacturing industry in Malaysia. But only 13500 tons are recycled, while the rest is incinerated or landfilled which is the common solid waste management applied around the world. However, the decomposition and burning of these solid rubber wastes with polymer content of >80%, ash and calcium carbonate content of 10%, and curatives of 2% (Sulphur, antioxidants, accelerators, and ZnO) can pollute the land and air while also wasting a valuable resource for important applications (Devaraj et al., 2017). Diversifying rubber waste management into more useful materials is an important topic for this research.

1.3 Research Objectives

The overarching goal of this study is to develop an efficient adsorbent from readily available rubber waste from the rubber glove manufacturing industry to remove TC from an aqueous solution.

The objectives of this research work are:

i. To modify and characterize the rubber waste as polymeric adsorbent.

ii. To study the batch adsorption of TC by using modified rubber waste (MRW) under various variables such as adsorbent dosage, solution pH, initial TC concentration, contact time, and reaction temperature in an aqueous solution.

iii. To investigate the characteristics of kinetics, isotherms, and thermodynamics for the adsorption of TC on MRW.

iv. To evaluate the reusable potential of MRW by regeneration and reusability studies

1.4 Scope of Research

i. The first objective involved the preparation and modification of rubber waste derived from the rubber industry, through facile sulfonation reaction. The physicochemical properties of the rubber waste and modified rubber wastes were determined using Fourier Transform Infrared (FTIR) spectroscopy, Brunauer-Emmett-Teller (BET) method, Energy Dispersive X-ray (EDX) analysis, Field Emission Scanning Electron Microscopy (FESEM) and the point of zero charges (pH_{PZC}).

ii. To achieve the second objective, a series of adsorption tests, using a basic batch adsorption process, was performed under different reaction conditions for systematic efficacy assessment of the MRW in removing TC in an aqueous solution.

iii. The adsorption experimental data were then fitted to Langmuir and Freundlich adsorption isotherm model and the pseudo-first and pseudo-second kinetic order to evaluate the adsorption capacity and adsorption mechanism. The data were also analyzed using Van't Hoff plot to determine the adsorption thermodynamics data.

iv. The final objective included desorption tests to determine the best eluent for removing TC from the MRW's surface, as well as MRW reusability.

1.5 Thesis Organization

This thesis is structured into five chapters that begin with the introduction, followed by a literature review, then materials and methods before results and discussions, and ends with conclusions and recommendations. References and appendices come after the fifth chapter. Chapter 1 also covers the research background, problem statements, justification, objective, the scope of the study. Chapter 2 deals with a comprehensive review of pharmaceuticals as an environmental pollutant. It also covers the pharmaceutical pollutants removal technologies, adsorption studies and adsorption mechanism, the adsorbent used in pharmaceutical pollutants adsorption, a brief introduction of the rubber uses, wastes, and its application is included in chapter 2. Chapter 3 covers a description of materials and equipment used, the method employed for Modification of rubber waste and characterization, adsorption experiment, and data analysis. The fourth chapter presents the results and its thorough discussion and finding of the research. The final chapter provides the overall summary of the research finding and suggestions for further modification and improvement.

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