NANOSCALED ZERO VALENT IRON NATURAL ZEOLITES FOR REMOVAL OF ANTIBIOTICS FROM WASTEWATER

JIMMY LYE WEI PING

UNIVERSITI TEKNOLOGI MALAYSIA

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JIMMY LYE WEI PING

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ABSTRACT

Nowadays, antibiotics have emerged as new kinds of organic micropollutants found in conventional sewage wastewater treatment plants, indicating the failure of coagulation-flocculation in treating antibiotics-containing wastewater. It might lead to the disperse of antibiotic resistance, which has a massive impact on human health and economic consequences globally. In recent years, nanoscale zero-valent iron (NI) has shown promising results for wastewater remediation to remove various contaminants. Thus, natural zeolite-supported nanoscale zero-valent iron (NI-NZ) has been proposed as a novel alternative to remove tetracycline (TC) and oxytetracycline (OTC). Synthesised NI-NZ was characterised using nitrogen adsorption-desorption, scanning electron microscopy, energy dispersive spectroscopy, X-ray photoelectron spectroscopy, and X-ray diffraction. The performance of the TC and OTC removal using NI-NZ in batch adsorption was assessed. The preliminary removal performance of natural zeolite (NZ), NI, and NI-NZ was compared, and it was observed that NI-NZ had a better performance in removing TC (333.33 μmol g^{-1}) compared to OTC (285.71 μmol g^{-1}). Thus, NI-NZ was chosen as the adsorbent and TC as the target antibiotic for further tests involving other parameters. The effect of vital parameters comprising initial pH, adsorbents dosage, temperature, initial antibiotic concentration, and contact time was studied. The optimum condition was at pH 3, 30 °C, and 1 g L^{-1} NI-NZ with approximately 97% of TC removed. Besides, the adsorption of TC has been proven to follow Langmuir and Temkin isotherm models. Kinetically, the process was best suited in the pseudo-second-order (PSO) and Elovich kinetic model. Furthermore, the thermodynamic study indicated that the reaction at room temperature was exothermic and spontaneous. The usability of NI-NZ in the continuous flow process has also been evaluated by using a fixed-bed column. The column experiments demonstrated that the breakthrough time increased with decreased initial TC concentration and flow rate and increased bed height. The cumulative kinetic data were in good agreement with the PSO and Elovich kinetic models. The Thomas and Yoon-Nelson models exhibited the closest predicted dynamic profile with the experimental results for all test conditions regarding the breakthrough curve. The applicability of NI-NZ in the adsorption coagulation-flocculation (ACF) process was evaluated using a jar test to remove TC from simulated wastewater. The outcome was encouraging because NI-NZ in ACF enhanced TC elimination from 10% to 75% at pH 3 with 0.4 g L^{-1} NI-NZ. The Langmuir isotherm model and PSO kinetic model were the most suitable model to represent the adsorption of TC via ACF. In conclusion, NI-NZ was successfully synthesised in this study. It has a high prospect of becoming one of the most effective solutions to remediate antibiotics-polluted wastewater because of its high applicability in batch adsorption, continuous adsorption, and ACF processes.

ABSTRAK

Pada masa kini, antibiotik telah muncul sebagai bahan pencemar mikro organik yang dikesan di loji rawatan kumbahan konvensional atas kegagalan proses pengentalan dan penggumpalan dalam rawatan air kumbahan yang mengandungi antibiotik. Isu ini menyebabkan merebaknya rintangan antimikrob yang mempunyai kesan negatif terhadap kesihatan dan ekonomi di seluruh dunia. Beberapa tahun kebelakangan ini, besi bervalensi sifar pada skala nano (NI) telah menunjukkan keputusan yang meyakinkan untuk merawat pelbagai jenis bahan pencemar yang didapati dalam air kumbahan. Oleh itu, penyelidikan ini bertujuan untuk mengkaji keberkesanan gandingan besi bervalensi sifar pada skala nano dengan zeolit semulajadi (NI-NZ) dalam menyingkirkan tetrasiklin (TC) dan oksitetrasiklin (OTC). NI-NZ yang disistesis dicirikan dengan menggunakan penjerapan-nyaherapan nitrogen, mikroskop elektron imbasan, spektroskopi fotoelektron sinar-X, pembelauan sinar-X dan spektroskopi serakan tenaga. Penilaian awal dijalankan dengan membandingkan prestasi penyingkiran TC dan OTC secara kelompok melalui zeolit semulajadi (NZ), NI dan NI-NZ. Keputusan menunjukkan bahawa NI-NZ mempunyai prestasi lebih baik dalam menyingkir TC (333.33 µmol g^{-1}) berbanding dengan OTC (285.71 µmol g^{-1}). Oleh itu, NI-NZ dipilih sebagai penjerap dan TC sebagai sasaran antibiotik untuk ujian selanjutnya yang melibatkan parameter lain. Kesan parameter penting seperti pH awal larutan, dos penjerap, suhu, kepekatan awal antibiotik dan masa sentuh telah dikaji. Prestasi optimum berlaku dengan hampir 97% TC telah disingkirkan pada pH 3 dan 30 °C dengan menggunakan 1 g L^{-1} NI-NZ. Di samping itu, ia telah terbukti bahawa penjerapan TC mengikuti model isoterma Langmuir dan Temkin. Secara kinetik, proses ini paling sesuai dengan model kinetik pseudo tertib kedua (PSO) dan Elovich. Tambahan pula, analisis termodinamik menunjukkan bahawa proses ini adalah eksotermik dan spontan pada suhu bilik. Penggunaan NI-NZ dalam proses penjerapan berterusan juga telah dinilai dengan menggunakan turus lapisan tetap. Kajian turus menunjukkan bahawa masa bulus meningkat apabila kepekatan awal influen dan kadar aliran TC menurun dan apabila ketinggian lapisan meningkat. Data bertokok kinetik didapati diwakili oleh model kinetik PSO dan Elovich. Bagi lengkung bulus, model Thomas dan Yoon-Nelson menawarkan profil dinamik terdekat dengan hasil ujikaji untuk semua keadaan ujikaji. Proses hibrid penjerapan, pengentalan dan penggumpalan (ACF) telah dijalankan melalui ujian balang untuk menyingkir antibiotik dari air kumbahan simulasi. Dengan bantuan NI-NZ, proses ACF telah meningkatkan penyingkiran TC dari 10% hingga 75% pada pH 3 dengan menggunakan 0.4 g L^{-1} NI-NZ. Model isoterma dan model kinetik yang paling sesuai untuk mewakili penyingkiran TC melalui ACF adalah model Langmuir dan PSO. Kesimpulannya, NI-NZ mempunyai prospek yang tinggi untuk menjadi salah satu jalan penyelesaian yang berkesan untuk menyingkirkan antibiotik daripada air kumbahan kerana kebolehgunaannya yang tinggi dalam proses penjerapan kelompok, penjerapan berterusan dan ACF.

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LIST OF ABBREVIATIONS

ACF	-	Adsorptive Coagulation-Flocculation
BDST	-	Bed Depth Service Time
BOD	-	Biological Oxygen Demand
CEC	-	Critical Micelle Concentration
COD	-	Chemical Oxygen Demand
D-R	-	Dubinin-Radushkevic
EBRT	-	Empty Bed Residence Time
EDS	-	Energy Dispersive Spectroscopy
ER	-	Exhaustion Rate
ESBL	-	Extended-Spectrum Beta-Lactamase
FAU	-	Faujasite
FTIR	-	Fourier-Transform Infrared Spectroscopy
L-H	-	Langmuir-Hinshelwood
MOR	-	Mordenite
NAD	-	Nitrogen Adsorption-Desorption
NB	-	Nitrobenzene
NI	-	Nanoscale Zero-Valent Iron
NI-NZ	-	Natural Zeolite-Supported Nanoscale Zero-Valent Iron
NZ	-	Natural Zeolite
OMC	-	Ordered Mesoporous Carbon
OTC	-	Oxytetracycline
PAC	-	Powdered Activated Carbon
PFO	-	Pseudo-First-Order
PNEC	-	Predicted-No-Effect Concentration
PSO	-	Pseudo-Second-Order
R-P	-	Redlich-Peterson
SEM	-	Scanning Electron Microscopy
TC	-	Tetracycline
UV	-	Ultraviolet
UV-Vis	-	Ultraviolet-Visible

WWTP - Wastewater Treatment Plant

XPS - X-Ray Photoelectron Spectroscopy

XRD - X-Ray Diffraction

LIST OF SYMBOLS

a - Elovich constant related to initial adsorption

a_{RP} - Redlich-Peterson constant

a_S - Sips constant

A_c - Clark constant

b - Elovich constant related to desorption

b_T - Temkin constant related to heat of sorption

b_{TO} - Toth constant

B - Slope of Boyd plot

B_t - Boyd plot parameter

C - Intra-particle constant related to the boundary thickness

C_1 - First stage intra-particle constant related to the boundary thickness

C_2 - Second stage intra-particle constant related to the boundary thickness

C_e - Equilibrium concentration

C_o - Initial concentration or influent concentration

C_t - Concentration at time t or effluent concentration

D_p - Effective pore diffusion coefficient

D_s - Film diffusion coefficient

E - Adsorption energy

F - Ratio of adsorption capacity at time t to equilibrium adsorption capacity

F_v - Volumetric flow rate

h - Initial adsorption rate

k_1 - Lagergren rate constant of the first-order adsorption

k_2 - Rate constant of PSO kinetic model

k_a - Adsorption coefficient

k_{av} - Avrami constant

k_b - Bangham constant

k_r - Reaction rate constant

K_{BA} - Bohart-Adams constant

K_d - Sorption equilibrium constant

K_F - Freundlich constant

K_{FH} - Flory-Huggins constant

k_{int} - Intra-particle rate constant

k_{int1} - First stage intra-particle rate constant

k_{int2} - Second stage intra-particle rate constant

K' - Dimensionless sorption constant

K_L - Langmuir constant

K_{ow} - Octanol/water partition coefficient

K_{RP} - Redlich-Peterson constant

K_S - Sips constant

K_T - Temkin constant

K_{TH} - Thomas rate constant

K_{YN} - Yoon-Nelson constant

m - Mass of adsorbent

m_v - Mass of adsorbent per volume of solution

n - Avrami coefficient

n_F - Freundlich coefficient

n_{FH} - Number of occupied sites

n_{TO} - Toth exponent

N_b - Dynamic adsorption capacity

N_o - Saturation concentration

pH_{equi} - Equilibrium pH

pH_{ini} - Initial pH

pH_{pzc} - Point of zero charge pH

pK_a - Acid dissociation constant

q_{cal} - Theoretical adsorption capacity

q_e - Adsorption capacity at equilibrium

q_{exp} - Experimental adsorption capacity

q_m - Maximum adsorption capacity

q_t - Adsorption capacity at any time, t

q_t^* - Accumulated adsorption capacity at time t

Q_t - Amount of TC adsorbed at time t

r - Radius of a particle

r_o - Initial reaction rate

r_c - Clark constant

R - Gas constant

R^2 - Correlation of determination

R_L - Langmuir separation factor

S_{BET} - Specific surface area

t - Time

$t_{0.5}$ - Experimental time of 50% breakthrough

t_b - Breakthrough time

t_s - Saturation time

T - Temperature

v - Linear velocity

V - Volume of solution

V_t - Accumulated volume of effluent at time t

$V_{treated}$ - Total treated volume of adsorbate solution

Z - Bed height

α - Bangham constant

β - D-R constant

β_{RP} - Redlich-Peterson exponent

β_S - Sips exponent

ΔG - Changes in Gibbs free energy

ΔH - Changes in enthalpy

ΔS - Changes in entropy

ε - Polanyi potential

η - Removal efficiency

θ - XRD detector angle

θ_{FH} - Degree of surface coverage

τ - Theoretical time of 50% breakthrough

χ^2 - Chi-square error

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CHAPTER 1

INTRODUCTION

1.1 Research Background

Antibiotics are pharmacological products used to stop or slow the growth of microorganisms in animals and humans. Antibiotic consumption increased by 65% globally between 2000 and 2015, owing to high demand in the cattle industry and medical applications (Klein *et al.*, 2018). In 2010, India was the largest consumer of antibiotics, followed by China and the United States (Van Boeckel *et al.*, 2014). Antibiotics use per capita in high-income countries like the United States, Hong Kong, South Korea, and Singapore was high, but it has plateaued. On the other hand, developing countries, such as Brazil, China, West Africa, and Saudi Arabia have seen a sharp increase in antibiotic consumption per capita (Sriram *et al.*, 2021).

Even though antibiotics were one of the greatest medical inventions since the 1940s, little or no awareness of the footprint of antibiotics was established. It has been speculated that various kinds of antibiotics had managed to exist silently in the environment for the last 30 years. Only recently, the existence of antibiotics has been accurately determined thanks to advanced analytical technologies. For instance, various classes of antibiotics were detected in the water matrices of different countries, including Southern France (Feitosa-Felizzola and Chiron, 2009), Turkey (Onmaz *et al.*, 2015), Belgium (Vergeynst *et al.*, 2015), and Sweden (Grabic *et al.*, 2012). The leading cause of antibiotics in wastewater, livestock farms, and hospital discharge streams is that a large amount of antibiotics consumed is excreted unchanged via faeces and urine (Du and Liu, 2012).

Nevertheless, antibiotics in water and soil did not receive as much attention as common pollutants such as heavy metals, dyes, and pesticides in recent decades. It could be comprehended as the effect of persistence of antibiotics in the environment is not evident in the short term. Unfortunately, in the long run, the discharge of antibiotics in nature matrices directly results in the spread of dangerous bacteria resistant to antibiotics. For instance, the prolonged exposure to antibiotics caused *Escherichia coli* bacteria (*E. coli*) to produce extended-spectrum β-lactamase enzyme, which is resistant to various antibiotics (Alonso *et al.*, 2016). It will cause the failure of certain antibiotics to treat diseases that could be treated effectively in the past. For example, more than 19000 deaths per year caused by methicillin-resistant Staphylococcus aureus had been reported in the United States (Jean and Hsueh, 2011).

Moreover, several illnesses and even deaths related to antibiotic-resistant bacteria were revealed in Malaysia (Yuen, 2018). This information shows that the spread of antibiotic-resistant was not contained in a specifically developed region but has already emerged as a global issue. Besides, the prolonged occurrence of antibiotics in water matrices could damage the microbial community (Paumelle *et al.*, 2021). Upon realising the seriousness of the problem, five types of antibiotics were included in the surface watch list in 2018 (European Commission, 2018). The regulation was established as long-term exposure to antibiotics might cause humans to be infected by reluctant diseases due to the spread of antibiotic-resistant bacteria. Although there is no current regulation to limit the amount of antibiotics in environmental matrices, the discharge of antibiotics from sewage wastewater plants should be monitored to achieve ecological sustainability and eliminate public health threats.

It was reported that various types of antibiotics were detected worldwide, mainly at the effluent of wastewater treatment plants (Gao *et al.*, 2012a; Yang *et al.*, 2014; Behera *et al.*, 2011; Sari *et al.*, 2014). This solid fact has firmly revealed the incapability of conventional wastewater treatment plants (WWTP) in removing antibiotics. Generally, conventional WWTPs consist of coagulation-flocculation in primary treatment and biological removal in secondary treatment. However, it was reported that both treatment methods were not effective in removing antibiotics (Stackelberg *et al.*, 2007; Göbel *et al.*, 2007). Coagulation-flocculation is designed to remove turbidity and micropollutants with a log K_{ow} value > 5 (extremely hydrophobic) (Snyder *et al.*, 2003). Generally, the log K_{ow} values of antibiotics range from -2.92 to 3.8, justifying the ineffectiveness of coagulation-flocculation in removing antibiotics (Chen *et al.*, 2015). On the other hand, microorganisms used in secondary treatment might be affected by the toxicity of pollutants (Britto and Rangel, 2008).

A wide range of technologies has been studied to improve the removal of antibiotics in conventional WWTPs, which includes reverse osmosis (Alonso *et al.*, 2018), nanofiltration (Wang *et al.*, 2015), chlorination (Li *et al.*, 2014), ozonation (Lu *et al.*, 2020), ultraviolet disinfection (Ge *et al.*, 2015), and adsorption (Cheng *et al.*, 2021). The increased use of reverse osmosis and nanofiltration has been reported, but the membranes were prone to fouling and malfunction when they were in contact with oxidising agents (Homem and Santos, 2011). Chlorination was an alternative technique to treat antibiotics-containing wastewater. Still, there was a significant concern that the chlorinated by-products that might be formed were more dangerous than the antibiotics precursor (Li *et al.*, 2014).

Similarly, ozonation did not seem practical due to difficulty in pH control and the high possibility of forming biologically active chemical components (Homem and Santos, 2011). On the other hand, ultraviolet disinfection was less effective in antibiotic removal and required more energy than ozonation (Michael *et al.*, 2013). In contrast, adsorption was preferable in removing organic pollutants from the water as it did not generate harmful side products (Ali, 2013). It was reported that more than 90% of antibiotics could be removed by using activated carbon (Ahmed and Theydan, 2014). Despite the excellent performance of activated carbon, it is costly and challenging to regenerate (Larasati *et al.*, 2020).

Unlike activated carbon, natural zeolites exist abundantly in nature and are low-cost materials (Eroglu *et al.*, 2017). Zeolites are crystalline microporous aluminosilicates minerals that could be obtained naturally or synthetically. There are about 60 types of natural zeolites originating from the reaction between volcanic rocks and ash layers in the presence of alkaline water. It has been widely used as an adsorbent in the purification process due to its' high cation exchange capability. Natural zeolites have been proven effective in removing ammonium (Widiastuti *et al.*, 2011), heavy metals (Elboughdiri, 2020), and cationic dye (Korkmaz *et al.*, 2013). In contrast, the performance in the removal of antibiotics was not as good. However, natural zeolites could be easily modified to fit the removal needs.

In the last twenty years, nanoscale zero-valent iron (NI) emerged as a versatile alternative for treating soil and water as it has the advantages of large specific surface area and high reactivity (Johnson *et al.*, 2013). Also, it could be operated easily and was comparatively low in cost. Fruitful researches on the removal of several types of pollutants such as chlorinated contaminants (Su *et al.*, 2012), heavy metals (Li *et al.*, 2017a), nitrates (Hwang *et al.*, 2011a), and other inorganic compounds (Eljamal *et al.*, 2016) via NI had been conducted. It also had demonstrated good prospects for removing antibiotics (Xia *et al.*, 2014; Ahmed *et al.*, 2017).

Nevertheless, NI particles possess high surface energy and intrinsic magnetic interaction, which cause these nanomaterials to agglomerate into larger particles (Yan *et al.*, 2010). Therefore, it might reduce the stability and reactivity of NI. In order to solve the limitation, NI had been modified with various types of materials, including starch (Fu *et al.*, 2015), pumice (Guler, 2017), carbon spheres (Wang *et al.*, 2016c), and bentonite (Weng *et al.*, 2014). From that, varied performance on the removal of antibiotics had been observed. Nevertheless, alternative supporting material for NI could still be explored. Moreover, the research on the application and mechanism of modified NI on removing antibiotics is still rare.

1.2 Problem Statement

Fruitful research and development of antibiotics in reducing mortality rates of various diseases has resulted in large antibiotics consumption globally, especially in low-middle-income countries. For instance, there was about 50% increase in antibiotic consumption in Malaysia from 2001 to 2015 (CDC, 2018). Consumed antibiotics were partially metabolised and discharged via urine or faeces to the wastewater. However, conventional wastewater treatments were ineffective in degrading or removing antibiotics from wastewater (Rossmann *et al.*, 2014). In fact, most of the concentration of various antibiotics detected in the discharge of global WWTP exceeded the predicted-no-effect concentration (PNEC). One of the most frequently detected antibiotic classes in Asia was tetracycline which has concentration as high as 1536 g L^{-1} (Tran *et al.*, 2018).

Escaped antibiotics from the effluent of a conventional WWTP might reach surface water, groundwater, and even drinking water. The main problem caused by the discharge of antibiotics in the environment is the cultivation of resistant bacteria, which was recognised as one of the top ten threats to global health in 2019 (WHO, 2019b). It was estimated that the global death due to antibiotic resistance has already reached 700000 per year (WHO, 2019a). Leaving unattended, by 2050, the health and economic impacts were estimated to be 10 million annual human fatalities and a loss of 100 trillion USD (Regea, 2018). In order to resolve this severe issue, the discharge of antibiotics to the environment should be reduced by implementing effective wastewater treatment technologies.

The removal of antibiotics via adsorption has been evaluated and has gained some extent of success (Chao *et al.*, 2014). A study has proven that at least 90% of norfloxacin and ciprofloxacin were removed from the wastewater stream via activated carbon in an adsorption system (Ahmed and Theydan, 2014). However, activated carbon is costly, and it is challenging to regenerate as spent activated carbon needs to be transported to a specialised facility to perform the regeneration process (Larasati *et al.*, 2020). Thus, the search for more versatile adsorbents continues to be a challenge. On the other hand, a more cost-effective adsorbent such as natural zeolite (NZ) could be used as an adsorbent to remove pharmaceutical products such as antibiotics (Eroglu *et al.*, 2017). Furthermore, modifications of NZ have been done to achieve a better adsorption performance (Guo *et al.*, 2013).

Adsorbents have been commonly used in batch and continuous systems. However, it is challenging to filter spent adsorbent in a batch system and regenerate it in a continuous system. In order to overcome the challenge, a hybrid of adsorption and coagulation-flocculation process (ACF) has been introduced as a novel method in wastewater treatment. Adsorption and coagulation-flocculation processes occur at the same time after adsorbent and coagulant are simultaneously introduced in a mixing tank that contains wastewater. Besides improving the removal of contaminants and turbidity, spent adsorbent can be recovered for disposal (Kumari and Gupta, 2020). Thus, ACF is attractive in cost as a specific process unit is not required because exhausted adsorbents will be suspended at the bottom of the sediment tank via gravity due to coagulation-flocculation. Some researchers have successfully discovered the effectiveness of hybrid adsorption-coagulation in removing natural organic matter (Kang *et al.*, 2017), heavy metal (Wu *et al.*, 2013), and endocrine-disrupting chemicals (Joseph *et al.*, 2013).

Nevertheless, adsorbed contaminants require degradation to avoid secondary pollution. Recently, nanoscale zero-valent iron (NI) has been proven to degrade various organic and inorganic pollutants, including antibiotics (Xia *et al.*, 2014; Fang *et al.*, 2011). However, NI has a strong tendency to agglomerate, affecting its reactivity and stability. To overcome agglomeration, researchers have tried to immobilise NI on various materials such as alumina (Yesiller *et al.*, 2013), chitosan (Horzum *et al.*, 2013), and natural zeolite (Nairat *et al.*, 2015) for the removal of rare earth elements, inorganic arsenic, and dye, respectively.

Natural zeolite is an abundant and inexpensive adsorbent, and therefore, it is highly intriguing to utilise it as a supporting material for NI to prevent agglomeration. The main focus of the present study is to develop a novel natural zeolite-supported nanoscale zero-valent iron (NI-NZ) and examine the versatility of the adsorbent in removing antibiotics using batch adsorption, continuous adsorption, and adsorptive coagulation-flocculation (ACF) processes. The use of NI-NZ for antibiotic elimination via batch, continuous adsorption, and ACF has not been documented to date. Although coagulation-flocculation is ineffective in removing antibiotics, the combined process achieved the synergistic effect by reducing the turbidity in antibiotic-containing wastewater, settling down the exhausted adsorbent, and, most importantly, removing and degrading antibiotics.

1.3 Research Objectives

According to the issues mentioned earlier, this study sets out with the following objectives:

1. To synthesise and characterise natural zeolite supported nanoscale zero-valent iron (NI-NZ).
2. To analyse the antibiotic removal via batch and continuous adsorption.
3. To evaluate the antibiotic removal performance via adsorptive coagulation-flocculation (ACF).

1.4 Research Scopes

The scopes of research have been identified and are mentioned below:

For Objective 1:

NZ, synthesised NI, and NI-NZ were characterised to study several essential features of these materials. First, physical properties such as the specific surface area, pore diameter, and porosity of the adsorbents were determined using the nitrogen-adsorption-desorption (NAD) method. Second, the morphology of the adsorbents was studied via scanning electron microscopy (SEM). Third, the adsorbents' mineralogy and element composition were analysed using X-ray diffraction (XRD) and energy dispersive spectroscopy (EDS). Finally, the X-ray photoelectron spectroscopy (XPS) method was used to study the type of iron species found on NI-NZ before and after treating the antibiotic solution.

For Objective 2:

Tetracycline, which is one of the most common and persistent antibiotics, was selected as the target antibiotic in this project. For batch adsorption study, important adsorption parameters such as pH (3-9) and dosage (0.2-1.8) g L^{-1} were individually studied. In addition, the temperature was varied (30, 50, 70) °C to compute the standard Gibbs free energy change, standard enthalpy change, and standard entropy change to evaluate the thermodynamic of the adsorption. Furthermore, the antibiotic adsorption isotherm and kinetics were evaluated. Adsorption isotherm data were fitted into fundamental isotherm models: Freundlich, Langmuir, Dubinin-Radushkevich, and Temkin models to determine the nature of adsorption and estimate the maximum adsorption capacity. Existing adsorption kinetic models, including pseudo-first-order (PFO), pseudo-second-order (PSO), Elovich, and intra-particle models, were used to fit kinetic data for adsorption kinetic study.

A single fixed-bed adsorption column was used for continuous adsorption of antibiotics. The performance of adsorption was evaluated according to the concept of the breakthrough curve, where vital variables for continuous adsorption, including flow rate (3, 5, 7) mL min^{-1}, bed height (0.4, 0.5, 0.6) cm, and initial concentration of antibiotics (10, 20, 40) μM were varied. Similarly, kinetic data were fitted into existing adsorption kinetic models, consisting of PFO, PSO, Elovich, and intra-particle models. Besides, continuous adsorption models, namely Bohart-Adam, Thomas, Yoon-Nelson, and Clark models, were used to evaluate the performance of NI-NZ adsorbent in a fixed-bed adsorption column.

For Objective 3:

The combined process of adsorption with coagulation-flocculation was applied to remove antibiotics from simulated wastewater via NI-NZ as an adsorbent. Performance determining factors such as pH (3-11), NI-NZ dosage (0.02-0.16) g L^{-1}, initial antibiotic concentration (5-700) μM, and mixing time (5-90) min was varied to study these parameters' effect on the antibiotic and turbidity removal. The mechanism of ACF was proposed using Langmuir, Freundlich, Dubinin-Radushkevich, and

Temkin isotherm models. Moreover, PFO, PSO, Elovich, and Weber-Morris intra-particle models were used to study ACF's kinetics.

1.5 Significance of Study

Antibiotic resistance is a very severe global issue caused by the accumulation of discharged antibiotics from conventional WWTPs. Effective and economical-viable solutions to improve current treatment technology in remediating antibiotics-containing wastewater is still being sought. This research explored the possibility of applying the novel NI-NZ in batch, continuous adsorption, and adsorptive coagulation-flocculation to remove and degrade antibiotics in the wastewater, preventing the spread of antibiotic-resistant bacteria. The findings of this study will provide insight into the removal mechanism and benefit the national and international wastewater treatment plants, eventually, the health of global citizens.

1.6 Thesis Outline

This thesis comprises five chapters. Research background, problem statement, objectives, scopes of study, research proposal outline, and summary can be found in Chapter 1. Chapter 2 includes the literature review in recent years such as antibiotics removal technologies, application of NI and supported NI in wastewater treatment, removal of antibiotics via adsorptive coagulation-flocculation. The last part of the chapter clarifies the research gap that could be filled in this study. Research methodology, including the preparation and synthesis of adsorbents, batch adsorption, continuous adsorption, and adsorptive coagulation-flocculation experiments procedures, were stated in Chapter 3. Besides, the procedures of characterisation of NI, NZ, and NI-NZ are explained in this chapter. Chapter 4 displays the experimental results of characterisation, batch adsorption, fixed-bed adsorption, and adsorptive coagulation-flocculation. The effect of various essential variables on the removal of antibiotics is discussed and evaluated. Last but not least, Chapter 5 concludes the findings of this research, and possible recommendations for future works are suggested at the end of the chapter.

1.7 Summary

The prolonged existence of antibiotics in water matrices and the environment has raised concerns that the balance of aquatic biodiversity and human health will be unwittingly affected. Typical WWTPs could not remediate emerging pollutants such as antibiotics. Therefore, adsorptive coagulation-flocculation (ACF) was proposed as a novel wastewater treatment technology to solve this global issue of the decade. The adsorbent for ACF was synthesised by impregnating nanoscale zero-valent iron (NI) on natural zeolite (NZ) due to the excellent reducing characteristic of NI and stability of NZ to be used as support to prevent the agglomeration of NI. The adsorption isotherm and kinetics were evaluated. The influence of some critical parameters such as pH, temperature, adsorbent dosage, contact time, and initial concentration of antibiotics was studied by carrying out batch adsorption. In addition, the suitability of NI-NZ to be used as an adsorbent in a fixed-bed adsorption column was evaluated. In the next phase, prior to application in industry, a jar test was conducted to mimic ACF to remove the antibiotic from simulated wastewater.

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LIST OF PUBLICATIONS

Journal with Impact Factor

1. **Lye, J. W. P.**, Saman, N., Noor, A. M. M., Mohtar, S. S., Othman, N. S., Sharuddin, S. S. N., Kong, H., & Mat, H. (2020). Application of nanoscale zero-valent iron-loaded natural zeolite for tetracycline removal process. Chemical & Engineering Technology, 43, 113. https://doi.org/10.1002/ceat.201900479. **(Q2, IF:2.418)**

2. **Lye, J. W. P.**, Saman, N., Sharuddin, S. S. N., Othman, N. S., Mohtar, S. S., Noor, A. M. M., Buhari, J., Cheu, S. C., Kong, H., & Mat, H. (2017). Removal performance of tetracycline and oxytetracycline from aqueous solution via natural zeolites: an equilibrium and kinetic study. CLEAN Soil Air Water, 45(10), 1600260. https://doi.org/10.1002/clen.201600260. **(Q3, IF:1.442)**