SIMULTANEOUS REMOVAL OF AMMONIUM AND PHOSPHATE USING MAGNESIUM CHLORIDE MODIFIED ZEOLITE

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SIMULTANEOUS REMOVAL OF AMMONIUM AND PHOSPHATE USING MAGNESIUM CHLORIDE MODIFIED ZEOLITE

by

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LIST OF SYMBOLS

Symbols		Units
C	Interparticle diffusion model constant	$(\frac{mg}{g})$
C_e	Final nutrient concentration	$(\frac{mg}{L})$
C_i	Initial nutrient concentration	$(\frac{mg}{L})$
k_1	Adsorption rate constant for pseudo-first-order model	$(\frac{1}{hr})$
k_2	Adsorption rate constant for pseudo-second-order model	$\frac{g}{(\frac{mg}{h})}$
k_3	Adsorption rate constant for interparticle diffusion model	$\left(\frac{\frac{mg}{g}}{h^{\frac{1}{2}}}\right)$
K_F	Sorption equilibrium constant for Freundlich model	$(\frac{mg}{g})(\frac{L}{mg})^{\frac{1}{n}}$
K_L	Sorption equilibrium constant for Langmuir model	$(\frac{L}{mg})$
n	Dimensionless Freundlich linearity constant	
q_e	Amount of adsorbed nutrient at equilibrium	$(\frac{mg}{g})$
q_m	Maximum sorption capacity	$(\frac{mg}{g})$
q_t	Amount of adsorbed nutrient at time, t	$(\frac{mg}{g})$
RE	Removal efficiency	%
t	Reaction time	min
V	Volume of aqueous solution	L
W	Adsorbent weight	g

LIST OF ABBREVIATIONS

Al	Aluminum
Al^{3+}	Aluminum ion
Ca	Calcium
Ca^{2+}	Calcium ion
EDX	Electron Dispersive X-ray Spectrometer
Fe	Iron
Fe^{3+}	Ferric ion
H^{+}	Hydrogen ion
H_2SO_4	Sulphuric acid
H_3PO_4	Phosphoric acid
HCl	Hydrochloric acid
HNO_3	Nitric acid
K	Potassium
K^{+}	Potassium ion
KH_2PO_4	Potassium dihydrogen phosphate
Mg	Magnesium
Mg^{2+}	Magnesium ion
$MgCl_2$	Magnesium Chloride
N	Nitrogen
Na	Sodium
Na^{+}	Sodium ion
Na_2CO_3	Sodium carbonate
NaCl	Sodium chloride
$NaHCO_3$	Sodium bicarbonate
$NaHCO_3/Na_2CO_3$	Sodium carbonate/sodium bicarbonate buffer solution
NaOH	Sodium hydroxide
NH_4^{+}	Ammonium ion
NH_4Cl	Ammonium chloride
O	Oxygen
OH^{-}	Hydroxide ion
PO_4^{3-}	Phosphate ion

SEM	Scanning Electron Microscope
Si	Silicon
Si^{4+}	Silicon ion
ZSM-5	Zeolite Socony Mobil-type 5

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APPENDIX A Calibration curve

PENYINGKIRAN SERENTAK AMONIUM DAN FOSFAT MENGGUNAKAN ZEOLIT YANG DIUBAH SUAI MAGNESIUM KLORIDA

ABSTRAK

Kajian ini menganalisis penyerapan amonium (NH_4^+) dan fosfat (PO_4^{3-}) secara individu dan serentak dengan mekanismanya bersama parameter operasi utama seperti kepekatan nutrien awal, jenis zeolite dan penambahan garam ke zeolit yang dimodifikasi Mg^{2+}. Fosfat melaporkan kecekapan penyingkiran sebanyak -62.68 % bagi penyerapan secara individu dan 13.49 % bagi penyerapan secara serentak. Selain itu, kecekapan penyerapan amonium pada penyerapan individu dan serentak masing-masing adalah 59.98 % dan 35.87 %. Kecekapan penyingkiran amonium dan fosfat menurun apabila kepekatan nutrien awal meningkat. Seterusnya, zeolit yang ditaip mordenite menunjukkan penyerapan yang lebih baik secara individu dan serentak dengan kecekapan penyingkiran yang lebih tinggi 59.98 % dan 35.85 %, masing-masing untuk amonium pada kepekatan awal 20 ppm. Walau bagaimanapun, zeolit-Y mempunyai kapasiti penjerapan lebih banyak ke arah fosfat dengan kecekapan penyingkiran yang lebih tinggi sebanyak 19.91 %. Kehadiran Na^+ ke dalam sistem reaksi mempunyai kesan negatif yang signifikan terhadap penyingkiran amonium kerana Na^+ mempunyai radius ion yang kecil berbanding ammonium dan mengurangkan kapasiti penyerapan. Penjerapan nutrien dapat dijelaskan dengan baik oleh model pseudo tertib kedua dengan (R^2 = 0.99) di mana prosesnya diatur oleh tindakan kimia. Bagi fosfat, sistem reaksinya dapat dijelaskan oleh model penyebaran intrapartikel dengan (R^2 = 0.89) di mana prosesnya dikawal oleh penjerapan permukaan luaran. Sebaliknya, kajian lengkung sesuhu mendedahkan lengkung suhu Langmuir sesuai untuk ciri penjerapan amonium dengan pekali korelasi yang tinggi

(R^2 = 0.995) menunjukkan bahawa penjerapan adalah berlapis mono. Penambahan $MgCl_2$ ke sistem reaksi telah mempromosikan proses penyahpisiran bagi kedua-dua sistem individu dan serentak menunjukkan kemungkinan penjanaan semula dan penggunaan semula bahan penjerap.

SIMULTANEOUS REMOVAL OF AMMONIUM AND PHOSPHATE USING MAGNESIUM CHLORIDE MODIFIED ZEOLITE

ABSTRACT

This study analyzed individual and simultaneous sorption of ammonium (NH_4^+) and phosphate (PO_4^{3-}) and their mechanisms together with their major operating parameters such as initial nutrient concentration, types of zeolites, and addition of salt onto Mg^{2+}-modified zeolites. The phosphate reported a removal efficiency of -62.68% due to poor sorption for individual sorption, and 13.49% for simultaneous removal. Besides, the removal efficiency of ammonium in individual and simultaneous sorption is 59.98% and 35.87%, respectively. The ammonium and phosphate removal efficiency decreases as the initial nutrient concentration increases. Moreover, the mordenite typed zeolites showed much better adsorption for both individual and simultaneous with a higher removal efficiency of 59.98% and 35.87%, respectively for ammonium at an initial concentration of 20 ppm. However, the zeolite-Y had more adsorption capacity towards phosphate with a higher removal efficiency of 19.91%. The individual presence of Na^+ had a significantly negative effect on the removal of ammonium as Na^+ possesses small ionic radii than NH_4^+, which reduces ammonium uptakes. The pseudo-second-order model could describe the adsorption of nutrients with higher ($R^2 \geq 0.99$), where chemical actions mainly govern the process. The Langmuir model can be acceptably applied to fit the experimental data with a higher correlation coefficient ($R^2 = 0.995$) for individual ammonium sorption, suggesting that the adsorption is a monolayer coverage. The addition of $MgCl_2$ to the reaction system promoting the desorption process for both separate and simultaneous processes revealed the possibility of regeneration and reusability of the adsorbent.

CHAPTER 1

INTRODUCTION

Nutrients such as ammonium and phosphate are essential for the growth of biological organisms. Still, excessive content of these nutrients in the lakes and river can lead to the eutrophication process and make the water toxic to the aquatic organisms. Therefore, these nutrients had to be removed and recover from swine wastewater, which can be used as fertilizer for the soil. This work proposes the zeolite-based adsorption method for the nutrient's removal and recovery where the adsorbent used is eco-friendly and could be utilized as fertilizer. Moreover, this study provides a sustainable cities and communities in the future who able to utilize all the available resources to fulfil their daily needs.

Chapter 1 introduces the overview of this research and the significance of the adsorption process for nutrient's removal and recovery. In general, this chapter summarizes the research background of the adsorption process and application of the adsorption process for simultaneous removal and recovery of ammonium and phosphate from wastewater, problem statement and objective of this final year project.

1.1 Research Background

Nitrogen and phosphorus are considered essential nutrients for the growth of biological organisms. However, an immoderate discharge of these components into the water bodies such as lakes and rivers will contribute to eutrophication, resulting in excessive growth of phytoplankton and fish toxicity together with the depletion of dissolved oxygen. Unfortunately, this can affect the drinking water system and human health (Xu et al., 2020). Thus, removing and recovering ammonium and phosphate from

contaminated water simultaneously is essential to prevent water pollution and to supply drinking water with excellent quality.

Therefore, a great effort has been devoted to developing low-cost and efficient technology to remove and recover the amount of ammonium and phosphate from wastewater, especially the methods to simultaneously adsorb both ammonium and phosphate. Many methods have globally existed to remove ammonium and phosphate globally such as crystallization, biological removal, precipitation, and ion exchange. Amongst the methods, adsorption is the most favourable method because it is relatively easy to use, less energy consumption, and is economical (He et al., 2016), especially when the nutrients are present in trace quantities such as aquaculture effluent. On the other hand, precipitation is considered less favourable due to the high operating cost and greatly influenced by the reagents cost such as alkali and magnesium (Xia et al., 2016). Usually, the amount of phosphate and magnesium in wastewater is insufficient, and significant amount of these salts are needed for the effective removal of ammonium which ultimately hinders the worldwide technology of this process (Huang et al., 2014).

Biological methods are often less favourable for removing nutrients since they did not respond well to shock loads of ammonium, and the unacceptable peaks of ammonium (NH_4^+) over the removal levels may regularly appear in the sewage. Moreover, inadequate generation, high operating cost, and unpredictability in outcome are drawbacks of biological-based methods. Besides, there is a high risk to safety during the process, such as the aeration process, which can cause stripping effects of volatile compounds resulting in accidental issues and health implications due to odour and aerosol releases (Alshameri et al., 2014). Compared to the methods mentioned above,

low operating cost, high safety, and relative ease of application and operation are some of the ascribes that cause an increased demand for the adsorption process to remove and recover ammonium and phosphate from contaminated water simultaneously.

Adsorption is the adhesion of atoms, ions, or molecules from gas, liquid, or dissolved solids to the surface, where it will create a film of the adsorbate on the surface of the adsorbent. So far, many researchers (He et al., 2016) have contributed to the development of effective adsorbents for the removal of ammonium and phosphate, where these components mainly consists of industrial by-products, natural minerals, synthetic materials, and agricultural wastes such as activated carbon, clay minerals, zeolites, fly ash, chitosan beads and oxide nanoparticles. Although several types of materials exist globally, removing ammonium and phosphate simultaneously and at low concentrations remains a significant challenge for many researchers. According to that, zeolites have been widely studied for the removal of ammonium.

Zeolites are microporous aluminosilicate minerals commonly used as catalysts and adsorbents (Huang et al., 2014). Due to its low market price and relative simplicity of operation and application, zeolite is gaining attention worldwide. In addition, zeolite generally exhibits high selectivity for removing ammonium and is often used as an ion exchanger in domestic water purification. Since natural zeolites possess lower adsorption capacity, it is generally being modified to increase adsorption capacity and the simultaneous removal of ammonium and phosphate from contaminated water. Whist many modifiers are available globally, such as acid treatment, iron oxide, and hydrated aluminium oxide, magnesium chloride modification is advantageous due to its eco-friendliness, cost-effectiveness, and biocompatibility.

The aquaculture industry is one of the industries that release nutrient-rich waste into the body of water. To ensure the sustainability of the aquaculture industry, the nutrient removal scheme should be replaced with the recovery mode for its further usage as fertilizer. In the recovery of ammonium and phosphate for fertilizer, the desorption process plays an important role. The uptake of the nutrients by the plant is closely related to the rate of nutrient desorption. The desorption process is carried out to regenerate the zeolite, recover the ammonium and phosphate, and get concentrated ammonium and phosphate effluent. The loaded zeolite can be utilized as fertilizer after a separation process done by filtration after the sorption desorption step. This is mainly done by treating the laden zeolite with hydroxides such as sodium hydroxide, potassium hydroxide, sodium bicarbonate, etc. These hydroxides will activate the zeolite surface by increasing the internal porous and later specific surface.

1.2 Problem Statement

To reduce the concentration of ammonium and phosphate from contaminated water, the simultaneous removal of ammonium and phosphate using zeolite is essential. Although both ammonium and phosphate must be removed from the dirty water, the simultaneous removal primarily by single material is challenging. Basically, two different kinds of reagents are required to pull cation (NH_4^+) and anion (PO_4^{3-}). For example, the application of an anion exchanger or a cation exchanger is preferable (ZHANG et al., 2007). However, the ion exchanger remains in the treated water, and the cost is high as well. Thus, modification of the adsorbent (zeolite) is mainly required to overcome such scenarios. Zeolite is widely used to remove ammonium and phosphate from the aquaculture pond. Although many types of modification such as modification

with iron oxide, hydrated aluminium oxide, magnetic iron oxide nanoparticles are available globally, modification with magnesium chloride has garnered increasing attention to remove ammonium and phosphate simultaneously mainly due to the method's eco-friendliness, cost-effectiveness, and biocompatibility. Furthermore, the modification of zeolite with magnesium chloride is considered more sustainable than the conventional treatment with natural zeolite as it can be used as a fertilizer amendment to the soil too after the treatment. Moreover, the chosen zeolite is eco-friendly to the soil and can desorb naturally within the ground, making it the best adsorbent globally. From a previous study, the removal of ammonium and phosphate is proven effective after modifying zeolite with magnesium chloride (Hao Wang et al., 2016). However, the simultaneous removal of ammonium and phosphate using magnesium chloride modified zeolite is still a challenging factor. Hence, the objectives of this study are to study the removal of ammonium and phosphate simultaneously for time effective factor using magnesium chloride modified zeolite which can be used as fertilizer later on, and different operating parameters were studied such as type of zeolite, initial concentration of nutrient, and addition of salt on the removal efficiency.

1.3 Objectives

The objectives of this study are:

a. To modify the zeolite to enhance its adsorption capacity for both ammonium and phosphate.
b. To study the mechanism and kinetic of the sorption and desorption process.
c. To study effect of different operating parameters such as type of zeolite, initial concentration of nutrient and salt addition on the adsorption efficiency.

CHAPTER 2

LITERATURE REVIEW

2.1 Ammonium and Phosphate discharge from industry

Municipal, agricultural, and industrial activities give rise to the discharges of ammonium and phosphate into the environmental resources. This excessive accumulation of ammonium and phosphate that is released into the water can result in serious problems such as eutrophication of the water body, toxicity in aquatic animals in the water body, and depletion of dissolved oxygen (Kubar et al., 2021; Seruga et al., 2019; Wang et al., 2018). Therefore, the industry's side streams and waste materials should be utilized to recover nutrients that reduce the usage of traditional inorganic fertilizers (Myllymäki et al., 2020).

2.2 Various Ammonium and Phosphate removal or recovery method

Recovery of discharged nutrients such as ammonium and phosphate hold promise in alleviating the water contamination problem. There is various type of removal and recovery methods for ammonium and phosphate such as precipitation, ion exchange, adsorption process, membrane separation and others (Sengupta et al., 2015).

Firstly, ion exchange had used as a method to remove ammonium and phosphate from wastewater. Ion exchange involves interchanging a charged ion between a solution and an insoluble resin (ion exchange resin). In a study, Jorgensen & Weatherley. (2003) demonstrated the ammonium removal using powdered resin, where a removal efficiency of 96% was obtained. Dong et al. (2020) reported removing phosphate using a hybrid anion exchanger and brought a removal efficiency of 95%. Although higher removal efficiency was obtained for both ammonium and phosphate, a question mark still hangs over the simultaneous removal of ammonium and phosphate using ion-

exchange, and only a few studies were reported on this. Guida et al. (2019) investigated the ammonium and phosphate removal using demonstration-scale ion exchange consisted of a drum filter, zeolite, and hybrid anion exchanger for ammonium and phosphate removal, respectively, and achieved more than 95% removal efficiency. However, long-term operation, regenerant and reuse factors become drawbacks for this method (Samuela Guida et al., 2021).

Besides, precipitation is being used widely in recovering ammonium and phosphate from wastewater. Previously, Okano et al. (2013) demonstrated the usage of amorphous calcium silicate hydrates in recovering phosphate, and an efficiency of 82% was achieved. Besides, Hu et al. (2020) shows the recovery of ammonium using chemical precipitation and achieves an efficiency of 85.9%. Removal of excess nutrients and relatively simple operation makes it a promising agent for recovery purposes. However, alkalinity requirement and pH control, and the increment in sludge production become a drawback of this method. As the application of sludge for irrigation is still inapplicable due to the limitations in bioavailability (Bayuseno & Schmahl, 2020).

Moreover, in the case of ammonium and phosphate recovery, membrane separation could also be used as a promising method. Membrane-based separation has been utilized in many wastewater operations with ultrafiltration, microfiltration, reverse osmosis, and nanofiltration. In a study, flat sheet gas permeable membrane was used to recover the ammonium and demonstrated that ammonium recovery achieved an efficiency of 75.9% (He et al., 2020). Besides, phosphate is being recovered using three complementary membrane-based processes, namely, nanofiltration, diafiltration, and

Donnan dialysis, and achieved an efficiency of 90% (Noubli et al., 2020). Although membrane separation offers some advantages over the recovery process, such as the absence of secondary pollutants in the permeate and recovery is independent of fluid flow rate, fouling and high costs of membrane synthesis become a barrier for the application (Ye et al., 2017).

2.3 Ammonium and Phosphate removal via Adsorption

Adsorption is a surface phenomenon in which a gas, liquid, or gas-liquid mixture is attracted and adhered to the surface of a solid called adsorbent and creates attachment via physical or chemical bonds. As the adsorption process offers some benefits over another method, it has been chosen to remove and recover ammonium and phosphate. Adsorption is currently used for water treatment purposes in removing and recovering nutrients due to its high efficiency, low cost, ease of operation, the possibility of recovering the adsorbent, and the possibility to use adsorbent in solid form. Besides, the water treatment costs for adsorption are 5.0-200 US$/m^3, while other technologies costs between 10 to 450 US$/m^3, which makes adsorption a good process in nutrient recovery (Dotto & McKay, 2020; Pathy et al., 2021). Moreover, to have high adsorption capacity, the chosen adsorbent must fulfil all the needed requirements such as large porous surface area, thermostability to withstand high thermal energy, controllable pore structure, and low acid/base reactivity (Daifullah et al., 2004; Foo & Hameed, 2009).

2.4 Type of adsorbent for adsorption

Adsorbent is a substance that adsorbs an adsorbate that has relatively high selectivity towards the adsorbent. Therefore, adsorbate will adhere to the adsorbent's surface when they are in contact. The selection of different adsorbent possessed different properties is crucial in the adsorption process as it will affect the capacity of

the adsorbent. Activated carbon, clay minerals, zeolites, fly ash, chitosan beads, and oxide nanoparticles are examples of adsorbents and are used widely for adsorption (He et al., 2016).

In a study, Lürling et al. (2014) had analysed the adsorption of phosphorus using clay minerals as the adsorbent; however, the humic substances interfere and cause the adsorption process to be ineffective. Clay minerals are hydrous aluminium phyllosilicates and form in water and are being familiar to humans from the earliest days of civilization. In that, vermiculite is a class of natural 2:1 type layered clay minerals. It is considered cheap, eco-friendly, and widely available, making it one of the best adsorbents globally (Liu et al., 2011). Due to its low-cost characteristics, it has been commonly used in refractory, construction, soil amendment, and so on. Ina previous study, Huang et al. (2014) demonstrated the usage of exfoliated vermiculites in the adsorption of phosphate. Exfoliated vermiculites are the product of vermiculites after a heating process. It possesses extraordinary physical and chemical properties such as low bulk density, highly developed porous structure, good chemical stability, high heat insulation, and high cation exchangeable capacity. Approximately 2.15 mg P/g of phosphate adsorption capacity is obtained using the exfoliated vermiculites in their study after 12 hr of operation.

Besides, Kilpimaa et al. (2014) and Zhang et al. (2012) reported the adsorption of phosphate and nitrate using activated carbon as the adsorbent. Activated carbon is a versatile adsorbent due to its better adsorption properties and its crucial role on filter material for the removal of colour, odour, and tastes from gases and liquids, and it can be produced from biomass-based raw materials and various fossil fuels. Due to its high cost, activated carbon usage is limited, increasing demand to derive activated carbon