Removal of Ammonia Nitrogen from Aqueous Solution

Using Alternative Adsorbents

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ABSTRACT

The capacity of alternative adsorbents including rice straw, delignified rice straw, and zeolite A for ammonia nitrogen removal from aqueous solution was investigated. The adsorption experiments were carried out in batch system with initial ammonia nitrogen concentration of 0.5 mg-N/l, 1000 ml ammonia nitrogen solution for 1 g of adsorbent. Ammonia nitrogen concentration was measured by using Phenolhypocholite method. Experimental data indicated that rice straw and delignified rice straw cannot adsorb ammonia nitrogen. However, zeolite A provided rapidly adsorption capacity for ammonia nitrogen removal. The amount of ammonia nitrogen adsorbed by zeolite A was 0.256 mg/g at 1 minute after the experiment was initially conducted and slightly stable at 0.275 mg/g after 10 minutes. The adsorption experiments were carried out at 0.125, 0.25, 0.5, 1.0, 2.0, 4.0, and 8.0 mg-N/l of ammonia nitrogen concentration. The results indicated that zeolite A had the adsorption capacity of 0.037, 0.102, 0.294, 0.551, 1.153, 2.303, and 3.897 mg/g respectively at equilibrium time. Langmuir and Freundlish adsorption models were applied to describe the equilibrium isotherms and the isotherm constants were determined. The correlation coefficient values for ammonia nitrogen adsorption isotherm using zeolite A adsorbent were best fitted with Freundlich model based on the obtained determination coefficients (R^2) was 0.992. Therefore, it employed to describe the adsorption sites between zeolite A and ammonia nitrogen are multilayer adsorption.

Keywords: Absorption, Alternative adsorbent, Ammonia, Rice straw, Zeolite A

เรื่อง ศึกษาการดูดซับสารละลายแอมโมเนียไนโตรเจนด้วยตัวดูดซับทางเลือก

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บทคัดย่อ

งานวิจัยนี้ศึกษาการดูดซับสารละลายแอมโมเนียไนโตรเจนด้วยตัวดูดซับทางเลือก ได้แก่ ฟางข้าวดิบ ฟางข้าวที่ผ่านการขจัดลิกนิน และซีโอไลต์เอ ทำการทดลองกระบวนด้วยการดูดซับแบบกะ ใช้สารละลายแอมโมเนียไนโตรเจนที่ความเข้มข้น 0.5 มิลลิกรัมไนโตรเจน/ลิตร ปริมาตร 1000 มิลลิลิตร ต่อ 1 กรัมตัวดูดซับ วัดความเข้มข้นของแอมโมเนียไนโตรเจนหลังผ่านกระบวนการดูดซับด้วยวิธีฟีนอลไฮโปคลอไรด์ ผลการทดลองพบว่า ที่สารละลายแอมโมเนียไนโตรเจนความเข้มข้น 0.5 มิลลิกรัมไนโตรเจน/ลิตร ทั้งฟางข้าวและฟางข้าวที่ผ่านการขจัดลิกนินไม่สามารถดูดซับแอมโมเนียไนโตรเจนได้ แต่ซีโอไลต์เอสามารถดูดซับแอมโมเนียไนโตรเจนได้อย่างรวดเร็ว ที่เวลา 1 นาที สามารถดูดซับแอมโมเนียไนโตรเจนได้ 0.256 มิลลิกรัม/กรัม และเริ่มคงที่ที่เวลา 10 นาที โดยมีความสามารถในการดูดซับแอมโมเนียไนโตรเจน 0.275 มิลลิกรัม/กรัม ศึกษาความสามารถในการดูดซับสารละลายแอมโมเนียไนโตรเจนที่ความเข้มข้นต่างๆคือ 0.125, 0.25, 0.5, 1.0 2.0, 4.0, และ 8.0 มิลลิกรัมไนโตรเจน/ลิตร พบว่า ซีโอไลต์เอมีความสามารถในการดูดซับแอมโมเนียไนโตรเจนที่สภาวะสมดุลคือ 0.037, 0.102, 0.294, 0.551, 1.153, 2.303, และ 3.897 มิลลิกรัม/กรัม ตามลำดับ ศึกษาแบบจำลองแลงเมียร์และฟรุนดลิชเพื่อใช้ในการอธิบายการดูดซับแอมโมเนียไนโตรเจนของซีโอไลต์เอ พบว่าการดูดซับแอมโมเนียไนโตรเจนสอดคล้องกับแบบจำลองฟรุนดลิช โดยมีค่า R^2 = 0.992 จึงสรุปได้ว่ากระบวนการดูดซับแอมโมเนียไนโตรเจนด้วยตัวดูดซับซีโอไลต์เอเป็นการยึดเกาะบนพื้นผิวที่เกิดจากการดูดซับแบบหลายชั้น

คำสำคัญ: กระบวนการดูดซับ; ฟางข้าว; ซีโอไลต์เอ; แอมโมเนียไนโตรเจน

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CHAPTER I
INTRODUCTION

1.1 Background [1]

Ammonia is widely used in the chemical industries as a bleaching agent in the production of fertilizers, plastics and explosives. The discharge of ammonia from wastewater treatment and industrial processes has been an important environmental issue and has been gained major worldwide concerns. Besides, the production of commercial fertilizers and other industrial applications, natural sources of ammonia include the decomposition of organic waste matters, human and animal wastes, and gas exchange with atmosphere and food scraps from aquatic lives. Ammonia does not only cause pollution of lakes and seas, but its excess level from the recommended limit dangerously harms aquatic lives and other animals. Therefore, it is necessary to remove ammonia from wastewater before discharge to environment.

Various methods have been employed to eliminate ammonia from wastewater including physical methods such as reverse osmosis and distillation, chemical methods such as chemical precipitation. However, these methods have some limitations and disadvantages. Reverse osmosis method requires large amount of energy input to provide hydraulic pressure driving water through the membrane. Moreover, semipermeable membrane can be easily damaged since suspended solids in wastewater are high. Distillation method requires large amounts of energy and water during the removal process. Chemical precipitation method requires working with corrosive chemicals, increasing operator safety concerns.

Adsorption is one of the treatment process which is a highly-efficient technique and has been gained considerable attention for treating ammonia from aqueous solution due to low initial cost, flexibility and simplicity of design. Among adsorption processes for wastewater treatment, activated carbon is the most widely used due to its high adsorption capacity and high specific surface area. However, the high cost of activated carbon has encouraged interest in using cheaper raw materials [2].

In recent years, most of attention has been considered and focused on the use of alternative adsorbents to replace the conventional adsorbents. Various unwanted plant wastes such as water hyacinth (Uddin et al. 2007; Abdallah 2013), rice husk (Kadhin and Al-Seroury 2012), and waste tea (Lutfee 2010) had been reported. These works explored the possibility to develop the efficient agricultural wastes as low cost adsorbents. There are many advantages of using unwanted plant wastes as adsorbent for wastewater treatment including simple technique, low cost, uncomplicated processing required, highly adsorption capacity and free availability.

In this work, rice straw, delignified rice straw, and zeolite A have been considered as an adsorbent for ammonia removal process. Rice straw is an agricultural waste, low cost, abundance, reliability, and availability. The surface of rice straw normally contains a lot of pores. Thus, rice straw has the potential to be used as an adsorbent. Zeolite A has been reported as an adsorbent used to remove cations from aqueous solution. Zeolite A can be synthesized by silica extracted from local rice straw using a simple extraction method. Thus, zeolite A has been studied as a potential adsorbent for ammonia nitrogen removal. The influence of contact time and initial concentration were investigated. The experimental adsorption data were fitted into Langmuir and Freundlich equations to describe the adsorption isotherm [3].

1.2 Objective

To study the removal efficiency of ammonia nitrogen from aqueous solutions by using alternative adsorbents

1.3 Scope of Work

1.3.1 Prepare adsorbent from rice straw and delignified rice straw for ammonia removal process

1.3.2 Synthesize zeolite A as an adsorbent for ammonia removal process

1.3.3 Characterize the crystallinity of zeolite A using X-Ray Diffractometer (XRD)

1.3.4 Study the influence of contact time and initial ammonia nitrogen concentration on ammonia nitrogen adsorption capacity

1.3.5 Study the adsorption isotherm during the ammonia removal process using the obtained adsorbent from rice straw and zeolite A

1.4 Expected Outputs

1.4.1 Reduce the amount of ammonia toxic in wastewater

1.4.2 Further encourage interest in using alternative adsorbents for wastewater treatment

CHAPTER II
THEORY AND LITERATURE REVIEWS

2.1 Adsorption process [4]

Adsorption is the process through which a substance, originally present in one phase, is removed from that phase and accumulate at the interface between that phase and separate phase. Adsorption process can occur at any solid-fluid interface, including with gas-solid and liquid-solid interface. The material being adsorbed is called "Adsorbate". The solid material being used as the adsorbing particle is called "Adsorbent".

2.2 Types of adsorption [5]

Forces of attraction generally exist between adsorbate and adsorbent. These force of attraction can be due to Van de Waal forces which are weak attractive force and chemical bond which is strong attractive force. On the basis of attractive force existing between adsorbate and adsorbent, adsorption process can be classified into two types, including "Physical adsorption" and "Chemical adsorption".

2.2.1 Physical adsorption

Physical adsorption process takes place when the force of attraction existing between adsorbate and adsorbent is weak Van der Waal forc, include with London dispersion force and electrostatic force. Due to weak attractive force, physical adsorption generally provides low enthalpy of adsorption (less than 20 kJ/mol) and reversible adsorption process can readily occur.

2.2.2 Chemical adsorption

Chemical adsorption involves with a chemical between the surface of adsorbent and adsorbate. New chemical bonds are generated at the adsorbent surface. The strong interaction between the adsorbate and the substrate surface creates new types of electrical bonds. Chemical adsorption provides high enthalpy of adsorption (50-400 kJ/mol) and the process is irreversible.

Table 2.1 Differences between physical adsorption and chemical adsorption

Physical adsorption	Chemical adsorption
1. The forces operating are weak Van der Waal forces. 2. The heat of adsorption is low (less than 20 kJ/mol). 3. The process is reversible. 4. Physical adsorption does not require any activation energy. 5. Physical adsorption takes place at low temperature and decreases with the increasing of temperature. 6. Physical adsorption forms multimolecular layer.	1. The forces operating are chemical bonds (ionic or covalent bond). 2. The heat of adsorption is high (less than 50-400 kJ/mol). 3. The process is irreversible. 4. Chemical adsorption requires activation energy. 5. Chemical adsorption increases with the increasing of temperature. 6. Chemical adsorption forms unimolecular layer.

2.3 Adsorption isotherm [6]

Adsorption isotherm is a curve relating the equilibrium concentration of a solute on the surface of an adsorbent, q_e to the concentration of the solute in the liquid, C_e. The adsorption isotherm is the equation relating the amount of solute adsorbed onto the solid and the equilibrium concentration of the solute in solution at a range temperature as shown in equation 2.1.

$$q_e = \frac{(C_i - C_e)\mathrm{V}}{m} \qquad (2.1)$$

Where q_e = the amount of adsorbate onto unit mass of the adsorbent (mg/g)

C_i = initial concentration (mg/l)

C_e = concentration at equilibrium time (mg/l)

V = the volume of solution (l)

m = the amount of adsorbent used (g)

There are several models for predicting the equilibrium distribution. The following four models are most commonly observed.

1. Linear adsorption isotherm

Linear adsorption isotherm presented in equation 2.2 is the simplest model in that the amount adsorbate into unit mass of the adsorbent is represented to be proportional to the concentration at equilibrium time.

$$q_e = KC_e \tag{2.2}$$

Where q_e = the amount of adsorbate onto unit mass of the adsorbent (mg/g)

K = Henry's adsorption constant

C_e = concentration at equilibrium time (mg/l)

2. Langmuir adsorption isotherm [6]

Assumption of Langmuir adsorption presented in equation 2.3 and 2.4 is called Ideal Localized Monolayer Model. That is the surface of the adsorbent is in contact with a solution containing an adsorbate which is strongly attracted to the surface, the surface has a specific number of sites where the solute molecules can be adsorbed, the adsorption involves the attachment of only one layer of molecules to the surface.

$$Q = \frac{Q_m b C_{eq}}{1 + bC_{eq}} \tag{2.3}$$

$$\frac{C_{eq}}{Q} = \frac{1}{Q_m b} + \frac{C_{eq}}{Q_m} \tag{2.4}$$

Where C_{eq} = the concentration at equilibrium time (mg/l)

Q = adsorption capacity (mg/g)

Q_m = the maximum adsorption capacity (mg/g)

b = monolayer adsorption constant

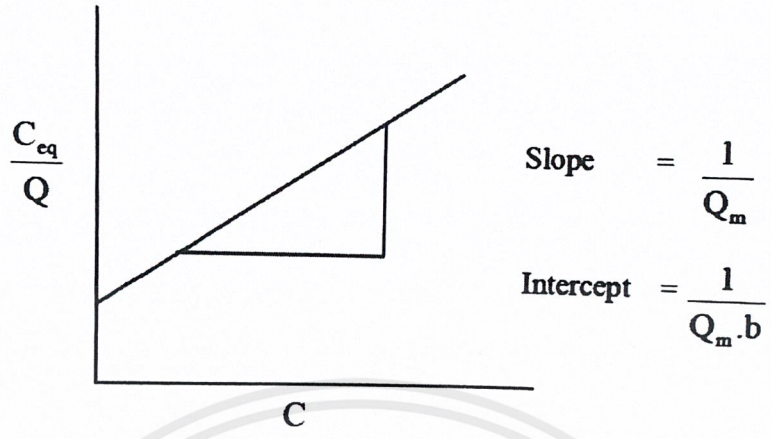


Figure 2.1. Linear Langmuir adsorption isotherm.

3. Freundlish adsorption isotherm [7]

The application of Freundlish adsorption isotherm presented in equation 2.5 and 2.6 generally explains exponential decay of adsorption energy onto adsorbent (Hameed and Daud, 2008).

$$Q = K_F C^{1/n} \tag{2.5}$$

$$\log Q = \log K_F + \frac{1}{n} \log C_{eq} \tag{2.6}$$

Where K_F = an indicator of adsorption capacity (mg/g)

1/n = a measure of intensity of adsorption

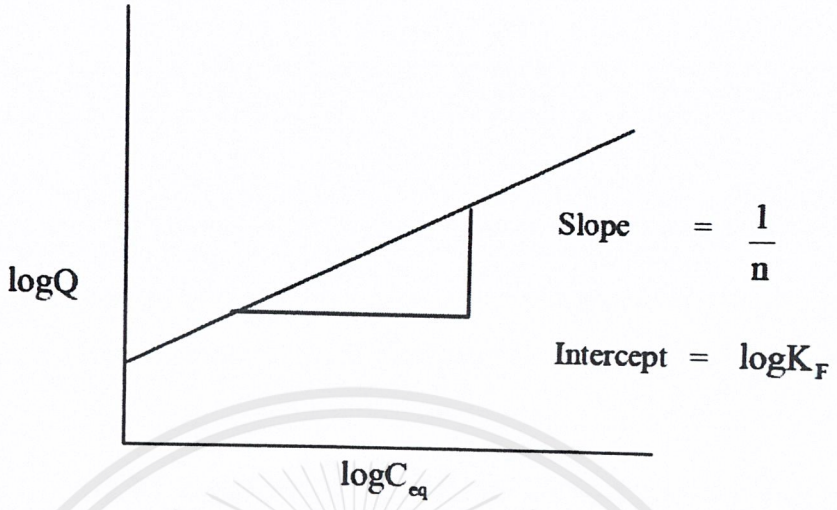


Figure 2.2. Linear Freundlich adsorption isotherm.

2.4 Ammonia in wastewater [8]

Ammonia is a colorless, pungent gaseous compound of hydrogen and nitrogen which is highly soluble in water. It is a biologically active compound found in most waters as a normal biological degradation product of nitrogenous organic matter (protein). Ammonia can be found its way to ground and surface waters through discharge of industrial process wastes containing ammonia and fertilizers. When dissolved in water, ammonia (NH_3) reacts to form ammonium (NH_4^+) and hydroxyl (OH^-) ions. When the pH is above 7.2, some free NH_3 remains and increases with increasing of pH. The equilibrium for these chemical species can be expressed by the following:

$$NH_3 + H_2O \leftrightarrow NH_4OH \leftrightarrow NH_4^+ + OH \qquad (2.7)$$

Oxidation of ammonia results in the formation of nitrite and nitrate.

2.5 Literature reviews

Sarker et al. 2015 [9] Removal of phenol from aqueous solution using rice straw as adsorbent. Three types of rice straw, including raw (untreated) rice straw, physically treated (boiled and dried) rice straw and thermally treated (heated at 230 °C for 3 hours to produce ash) rice straw, were investigated to determine phenol removal efficiency. Each type of rice straw was carried out by two sizes of particle (1 mm rice straw and <1 mm rice straw particles). The influence of various parameters, including different contact times and different adsorbent dosages, was considered. Colorimetric method was carried out to determine phenol concentration from aqueous solution. Langmuir and Freundlish adsorption isotherms were used to describe experimental data. Results were found that thermally treated <1 mm rice straw particle provided the highest phenol removal efficiency at 84% removal. The study of different contact times indicated that equilibrium time was around 4 hours and the maximum removal of phenol occurred within the first 1 hour. Phenol removal efficiency would increase as adsorbent doses increased. According to the difference type of rice straw, thermally treated (ash) rice straw provided the highest removal efficiency than raw rice straw and physically treated rice straw. This work indicates that three types of rice straw have the potential to be developed for removal of phenol from aqueous solution.

Wang et al. 2015 [10] The zeolite powder was carried out as adsorbent to investigate the performance of ammonia nitrogen adsorption. Zeolite powder, which is made of ground natural zeolite stone, was soaked in distilled water for 24 hours to remove very fine particles and died at 105 °C for 24 hours to remove moisture. Ammonia waste water was simulated through using ammonium chloride to make ammonia standard solution as waste water. XZ-0142 multipara meter was used to detect ammonia nitrogen concentration in aqueous solution. The effect of various experimental parameters on ammonia nitrogen removal was considered. The study on the effect of vibration time on adsorption capacity indicated that during different of contact times, the removal efficiency of ammonia nitrogen in aqueous solution was nearly constant. Contact time had little effect on the adsorption capacity of ammonia nitrogen. The equilibrium time occurred within 10

minutes. The study on the influence of reaction temperature on ammonia adsorption capacity indicated that in the range of 10-60 °C, the removal efficiency of ammonia nitrogen slightly decreased with increasing temperature. The study on the influence of initial concentration of ammonia solution on adsorption capacity indicated that the higher concentration, the greater adsorption capacity. This work indicates that zeolite powder is an effective adsorption for the removal of ammonia nitrogen from aqueous solution.

Ashraf A. et al. 2014 [11] Rice straw based carbon was investigated as low cost adsorbent for the removal of hazardous azocoumarin dye from aqueous solution. Rice straw based carbon (RSC) was modified by burning of waste rice straw in a special incinerator at the temperature of 1000-1200 °C. After selecting of adsorbent particle size through a 200 μm sieve and washing by distilled water, RSC was dried at 120 °C for 48 hours and prepared for the experiment. The Brunauer-Emmett-Teller (BET) was carried out to investigate pore size and pore volume of RSC. The BET surface area and the total pore volume of RSC were shown as 67.4 m^2/g and 0.134 cm^3/g respectively. Various parameters, including contact time, initial concentration, pH and temperature, were carried out to investigate the removal efficiency of hazardous azocoumarin dye using rice straw based carbon as adsorbent. The study of different initial concentrations indicated that when the initial dye concentration increased, the removal percentage of dye concentration decreased. The increasing of RSC dosage from 0.1 to 0.9 g was carried out to investigate the effect of adsorbent dosage. The result indicated that the higher dose of adsorbent, the more increasing of percentage of adsorption. The study of different temperature was investigated from 25 to 60 °C. The result showed a small increasing trend with a rise of temperature. The last observed factor is the effect of pH. The result indicated that the adsorption capacity of dye onto RSC increased significantly with decreasing pH. This work clearly demonstrates that rice straw based carbon (RSC) is an effective adsorbent for the removal of azocoumarin dye from aqueous solution.

Buasri A. et al. 2012 [12] The potential of rice straw as low cost adsorbent for Cu^{2+} removal was investigated. Various parameters, including contact time, initial concentration, pH and temperature, were carried out. Langmuir and Freundlish adsorption isotherms were used to describe experimental data. Results were found that rice straw is suitable and can be used as an effective adsorbent for Cu^{2+} removal from aqueous solution. The study of different contact times indicated that the adsorption of Cu^{2+} by rice straw had highly influenced by contact time. The amount of Cu^{2+} ions in aqueous solution decreased when contact time increased. The removal reached to equilibrium time at 120 min and the highest Cu^{2+} removal efficiency by rice straw occurred within first 30 min. The study of different initial concentration indicated that the amount of Cu^{2+} ions onto unit mass of rice straw at equilibrium increased rapidly at low initial Cu^{2+} concentration. Since initial Cu^{2+} concentration increased, the amount of Cu^{2+} ions adsorbed by rice straw could only slightly increase. The effect of pH indicated that the amount of Cu^{2+} ions adsorbed onto unit mass of rice straw at equilibrium increased by increasing of pH values. The highest removal efficiency of Cu^{2+} ions occurred at pH values between 9.0 to 11.0. The study of different temperature indicated that raising of temperature from 30 to 70 °C reduced the amount of Cu^{2+} in aqueous solution. This result indicated that using rice straw as adsorbent for Cu^{2+} removal is endothermic in nature.

Aydin et al. 2011 [13] The zeolite (clinoptilolite) was investigated the efficiency of ammonia nitrogen removal from landfill leachate. The zeolite was prepared by washing with distilled water to remove very fine particles and dying at 35-60 °C to remove moisture. Three types of adsorbent distinguished by treating with 1 N NaCl and 2 N NaCl and untreated zeolite were investigated adsorption capacity. The effect of different experiment parameters on removal of ammonia nitrogen from leachate was investigated. The study of different adsorbent dosage on the adsorption of ammonia nitrogen indicated that the removal efficiency increased with the increasing of adsorbent dosage. The zeolite treated by 2 N NaCl provided the highest adsorption capacity for ammonia nitrogen removal. Effect of contact time indicted that the rate of ammonia nitrogen increased rapidly during the first 30 min and remained nearly constant thereafter. After 12 hours, there was no change in ammonia nitrogen removal for every types of adsorbent. The equilibrium time was carried out at 1 hour. The study of different initial concentration indicated that the ammonia nitrogen removal capacity of zeolite increased with the decreasing of initial ammonia nitrogen concentration in landfill leachate solution. This work indicates that zeolite is an effective adsorbent for the removal of ammonia nitrogen from landfill leachate.

Table 2.2. Summary of literature reviews

Adsorbate	Adsorbent	Adsorption parameter	Reference
Phenol in aqueous solution	Rice straw - Raw - Physical treat - Thermal treat	- Contact time - Adsorbent dosage	[9]
Ammonia in aqueous solution	Zeolite powder	- Contact time - Temperature - Initial concentration of ammonia solution	[10]
Azocoumarin in aqueous solotion	Rice straw based carbon	- Contact time - Temperature - Initial concentration of ammonia solution - pH	[11]
Cu^{2+} in aqueous solution	Rice straw	- Contact time - Temperature - Initial concentration of ammonia solution - pH	[12]
Ammonia in landfill leachate	Zeolite (Clinoptilolite)	- Contact time - Temperature - Initial concentration of ammonia solution	[13]

CHAPTER III

RESEARCH METHODOLOGY

3.1 Experimental Equipment

1. 50 ml, 100 ml, 250 ml, and 1000 ml beaker
2. Glass rod
3. Reagent bottle
4. Analytical balance
5. Visible spectrophotometer (T60 VIS Spectrophotometer)
6. Centrifuge (GEMMY Model PLC-012 Series)
7. Incubator (RI Series BINDER REDLINE RI115)
8. Wash bottle
9. Magnetic stirrer (C-MAG HS 7)
10. 50 ml, 250 ml, and 1000 ml volumetric flask
11. Evaporating dish
12. Blender
13. Micropipette
14. Aluminium foil
15. Stand & clamp
16. Funnel
17. Condenser
18. Round bottom flask with joint
19. Rubber tube
20. Pump
21. pH test strips

3.2 Chemicals

1. Acetic acid
2. Ammonium sulfate
3. Sodium nitroprusside
4. Sodium hydroxide
5. Sodium chlorite
6. Sodium dichloroisocyanurate
7. Phenol
8. Trisodium citrate dihydrate
9. Sodium metasilicate

3.3 Preparation of Adsorbents

3.3.1 Rice straw adsorbent

1. Collect about 1 kg of fresh rice straw from the rice field in a polythene bag to avoid any contamination
2. Wash about 5 g of collected rice straw by distilled water to remove any contaminant
3. Soak washed rice straw in distilled water for 12 hours to initially remove color
4. Cut soaked rice straw into small pieces
5. Crush rice straw by using blender
6. Boil crushed rice straw in distilled water at 80°C for 3 hours and repeat this step for 3 times
7. Boil obtained rice straw from step7 in acetone at 50°C for 3 hours and repeat this step for 3 times. Cover beakers by aluminium foil to avoid evaporation of acetone
8. Dry obtained rice straw from step8 in incubator at 100°C, 24 hours
9. Preserve obtained rice straw in the air tight jar and keep a dry place

3.3.2 Delignified rice straw adsorbent [14]

1. Set up experimental equipment as shown in figure 3.1
2. Thoroughly wash about 5 g of collected rice straw by distilled water to remove any contaminant
3. Cut soaked rice straw into small pieces
4. Crush rice straw by using blender
5. Distill 14 ml of sodium chlorite ($NaClO_2$) in 250 ml of distilled water
6. Take 115 ml of solution from step 1 and adjust pH by acetic acid (CH_3COOH) until pH is about 4 (measure pH by using pH test strips)
7. Heat up the solution from step 6 in the round bottom flask (figure 2) until the temperature reaches to 70°C
8. Put and stir 3.5 g of rice straw in the heated solution using magnetic stirrer bar for 5 hours
9. Thoroughly wash obtained rice straw by distilled water
10. Preserve obtained rice straw in the air tight jar and keep a dry place

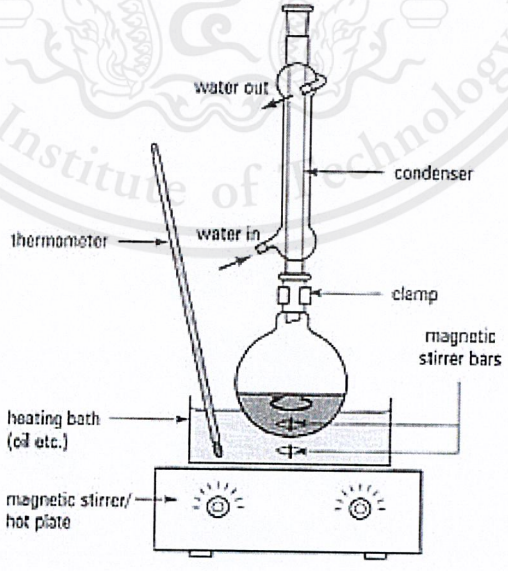


Figure 3.1. Reflux condenser setup for the modification of rice straw and zeolite A synthesis [15].

3.3.3 Zeolite A adsorbent [16]

1. Dissolve 8 g of sodium metasilicate by 44 g of distilled water using magnetic stirrer to employ the homogenous liquid mixture
2. Dissolve 3.4 g of sodium hydroxide in 34 g of distilled water
3. Add 6 g of sodium aluminate in the solution from step 2. Then, stir the solution using magnetic stirrer until the solution is clear
4. Gradually drop the solution from step 1 into the solution from step 3 using a dropper in order to create the molecular structure between alumina and silica. Then, mix the solution for 1 hour using magnetic stirrer
5. Setup the experimental equipment as shown in figure 3.1
6. Put and stir the solution from step 6 in the round bottom flask (figure 3.1), for 1 hour, 250°C
7. Pull the round bottom flask out of the joint and dip the bottom into water at room temperature
8. Separate obtained zeolite A and solution using the centrifuge. Repeat this step 2-3 times
9. Put obtained zeolite A on the watch glass covering with aluminium foil. Bake in the oven at 110°C for 24 hours
10. Preserve obtained zeolite A in the glass bottle

3.4 Preparation of Stock Standard Ammonia Solution

1. Dry 0.05892 of ammonia sulfate ($(NH_4)_2SO_4$) at 105-110 °C for 1-24 hours
2. Dissolve 0.05892 g of dried ammonia sulfate ($(NH_4)_2SO_4$) in 1000 ml in distilled water. Stock standard ammonia solution has a concentration of 12.5 mg-N/l
3. The stock solution is further diluted by distilled water to prepare test solutions of ammonia

3.5 Preparation of Chemical Reagents for Phenolnypochlorite Method [17]

3.5.1 Phenol Solution

Dissolve 5 g of phenol (C_6H_5OH) in 50 ml of 95% ethanol

3.5.2 Sodium Nitroprusside Solution

Dissolve 0.5 g of sodium nitroprusside (Na_2 ($Fe(CN)_5NO)\cdot 2H_2O$) in 100 ml of distilled water

3.5.3 Oxidizing Solution

Dissolve and mix 1 g of sodium dichloroisocyanurate ($C_3Cl_2N_3NaO_3$), 5 g of sodium hydroxide (NaOH) and 50 g of trisodium citrate dihydate ($Na_3C_6H_5O_72\cdot H_2O$) in 250 ml in distilled water

3.6 Preparation of Standard Curve of Ammonia Nitrogen Concentration

1. Dilute 0.5, 1, 2, 4, 8, 16, and 32 ml of ammonia solution (from 3.4) in 50 ml of distilled water separately. These ammonia solutions have a concentration of 0.125, 0.25, 0.5, 1, 2, 4, and 8 mg-N/l respectively.
2. Take 10 ml of ammonia solution from each concentration (from step 1)
3. Add 1 ml of phenol solution, 0.5 ml of oxidizing solution, and 1 ml of sodium nitroprusside solution respectively (from 3.5) to each ammonia concentration from step2. Then, mix thoroughly after each addition
4. Colors of each ammonia solution are allowed to develop at room temperature for 1 hour
5. The absorbance is related at 640 nm in visible spectrophotometer with a 10 cm length cuvette
6. Standard curve is contracted to correlate concentrations to different absorbance values

3.7 Characterize the crystallinity of zeolite A using X-Ray Diffractometer (XRD)

The crystallinity and X-ray diffraction (XRD) patterns of zeolite A were performed by X-ray diffractometer SHIMADZU XRD-6100. Operating conditions are shown below:

2Θ range of detection : 5 - 50°

Resolution : 0.02°

3.8 The Study of Ammonia Nitrogen Adsorption

Ammonia solution (from 3.4) is further diluted by distilled water to prepare test solution of ammonia

3.8.1 The study on the influence of initial concentration of solution on the adsorption capacity

1. Dilute 0.5, 1, 2, 4, 8, 16, and 32 ml of ammonia solution (from 3.4) in 50 ml of distilled water separately. These ammonia solutions have a concentration of 0.125, 0.25, 0.5, 1, 2, 4, and 8 mg-N/l respectively. Prepare 3 sets of ammonia solution
2. Add 1 g of rice straw to each set of ammonia solution
3. Shake thoroughly after each addition using magnetic stirrer
4. Collect samples from each set of experiment at 1, 3, 5, 10, 15, 20, 30, 60, 90, and 120 minutes respectively
5. Constant conditions are used throughout, namely, the agitation speed is 200 rpm, room temperature is carried out, and initial ammonia nitrogen concentration of each set is maintained constant.
6. Collect 10 ml of ammonia solution from each set of experiment
7. Add 0.5 ml of phenol solution, 1 ml of oxidizing solution, and 0.5 ml of sodium nitroprusside solution respectively to each collected sample of ammonia solution. Then, mix thoroughly after each addition
8. Colors of each collected sample is allowed to develop at room temperature for 1 hour

9. The absorbance is recorded at 640 nm in visible spectrophotometer with a 10 cm length cuvette.
10. Determine each concentration value from standard curve
11. Plot the relation between q_e with time
12. Repeat step 1-11 using 1 g of modified rice straw and 1 g of zeolite A adsorbent

3.8.2 The calculation of q_e and removal efficiency

$$qe = \frac{(Ci - Ce)\mathrm{V}}{m}$$

Where q_e = the amount of ammonia nitrogen adsorbed onto unit mass of the adsorbent (mg/g)

C_i = initial ammonia nitrogen concentration (mg/l)

C_e = ammonia nitrogen concentration at equilibrium time (mg/l)

V = the volume of ammonia solution (l)

m = the amount of adsorbent used (g)

$$\%\, removal = \frac{Co - Ct}{Co} \times 100$$

Where C_o = ammonia nitrogen concentration at the start of process (mg/l)

C_t = ammonia nitrogen concentration after time t (mg/l)

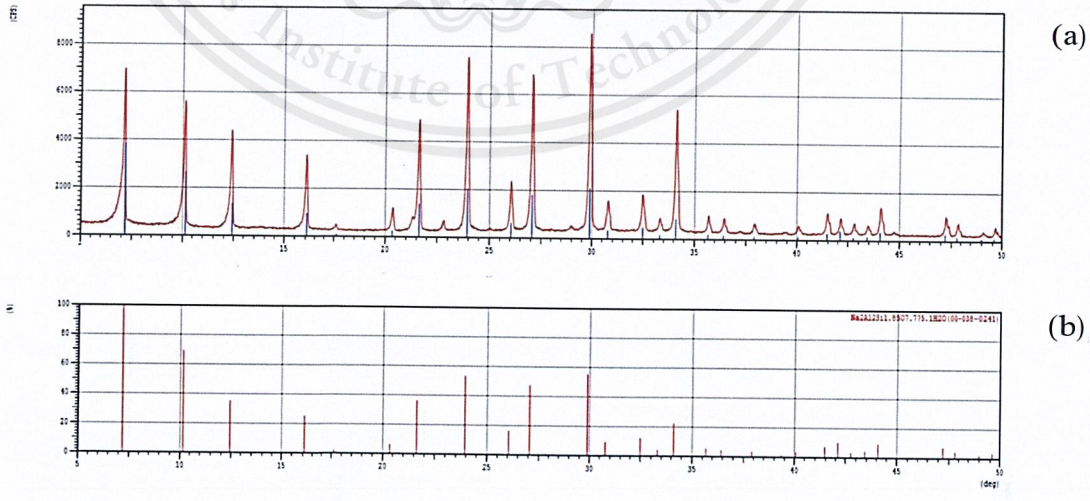
CHAPTER IV

RESULTS AND DISCUSSION

4.1 X-Ray Diffraction (XRD)

Zeolite A synthesis using sodium aluminate, sodium metasilicate, and sodium hydroxide was crystalized at 250 °C, 1 hour. The physical characteristic of obtained zeolite A presented in Figure 4.1 was white-fine powder. The overall crystalline phases of zeolite A were determined by XRD measurement. Radial scans of intensity recorded at ambient condition over 2θ angles indicated that the peak intensities were 7.14, 10.12, 12.40, 16.06, 21.62, 23.94, 27.06, 29.90, and 34.14 respectively matching with the data base of zeolite A as shown in Figure 4.2. Therefore, it could be confirmed that the sample characterized by XRD measurement was zeolite A.

Figure 4.1. Photograph of zeolite A powder.



(a)

(b)

Figure 4.2. The crystallinity of zeolite A using X-Ray Diffractometer (XRD): (a) obtained zeolite A (b) data base of zeolite A.

4.2 Effect of Different Adsorbents

Ammonia nitrogen removal of different adsorbents is presented in Figure. 4.1. Preliminary experiments were carried out to initially investigate the efficiency of rice straw, modified rice straw, and zeolite A adsorbents for ammonia removal with initial ammonia nitrogen concentration of 0.5 mg-N/l, 1000 ml ammonia nitrogen solution for 1 g of adsorbent. The experimental data indicated that ammonia nitrogen concentration remained constant after adsorption process using rice straw and modified rice straw adsorbents. However, the amount of ammonia nitrogen adsorbed by zeolite A was 0.256 mg/g at 1 minute after the experiment was initially conducted and slightly stable at 0.275 mg/g after 10 minutes. The high efficiency of zeolite A adsorbent is due to the high surface area of adsorption sites and the capacity to enhance the cation exchange with NH_4^+ from ammonia solution [13, 18].

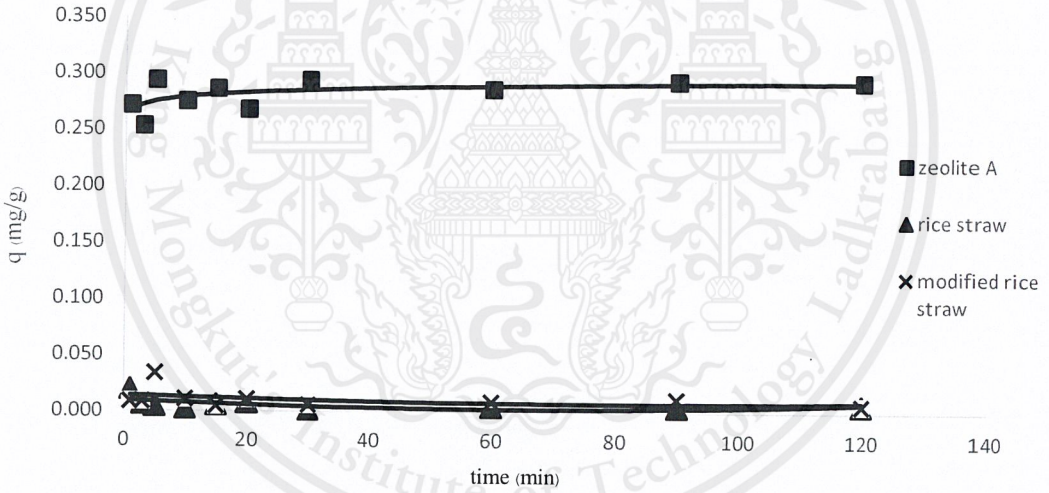


Figure 4.3. Ammonia adsorption capacity of rice straw, delignified rice straw, and zeolite A.

4.3 Effect of Initial Concentrations with Contact Time

The initial ammonia nitrogen concentration was determined and amount of adsorbed ammonia nitrogen in each set was calculated as a function of time. The adsorption experiments onto zeolite A adsorbent were carried out at 0.125, 0.25, 0.5, 1.0, 2.0, 4.0, and 8.0 mg-N/l of ammonia nitrogen concentration considering removal capacity at 1, 3, 6, 10, 15, 20, 30, 60, 90, and 120 minutes, using 1 g/l of zeolite A adsorbent. The rate of ammonia nitrogen removal was very rapid during the initial 1 minute and remained nearly constant thereafter for every initial concentration due to the number of vacant sites on the adsorbent were much available to adsorb the adsorbate molecules in the solution. The results indicated that zeolite A had the adsorption capacity of 0.037, 0.102, 0.294, 0.551, 1.153, 2.303, and 3.897 mg/g respectively at equilibrium time. Differences of initial ammonia concentration clearly showed that a gradual increase in the ammonia nitrogen concentration leaded to high adsorption capacity using zeolite A adsorbent since the more increased of ammonia nitrogen molecule in the solution, the higher driving force of ammonia nitrogen molecules adsorbed onto available sites of the zeolite A particles [19].

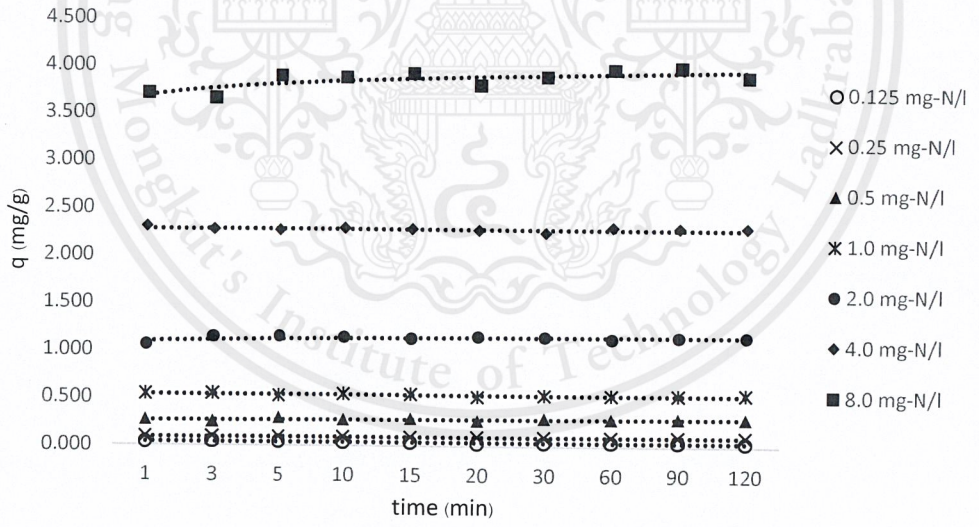


Figure 4.4. Effect of initial ammonia concentration on adsorption capacity using zeolite A adsorbent.

4.4 Adsorption Isotherms

The amount of ammonia nitrogen adsorbed onto unit mass of zeolite A adsorbent at equilibrium time (q_e) and the ammonia nitrogen concentration at equilibrium time (C_e) were calculated and correlated for the adsorption isotherm as shown in Figure 4.3. The corresponding correlation of adsorption capacity with equilibrium ammonia nitrogen concentrations after adsorption process conducted at different initial concentrations clearly indicated that the ammonia nitrogen removal capacity of zeolite A increased with the increased of ammonia nitrogen concentration. However, since the considering ammonia nitrogen concentration was low, the equilibrium capacity of ammonia nitrogen removal was not occurred. The maximum capacity of ammonia nitrogen adsorbed onto unit mass of zeolite A adsorbent was about 3.897 mg/g [7].

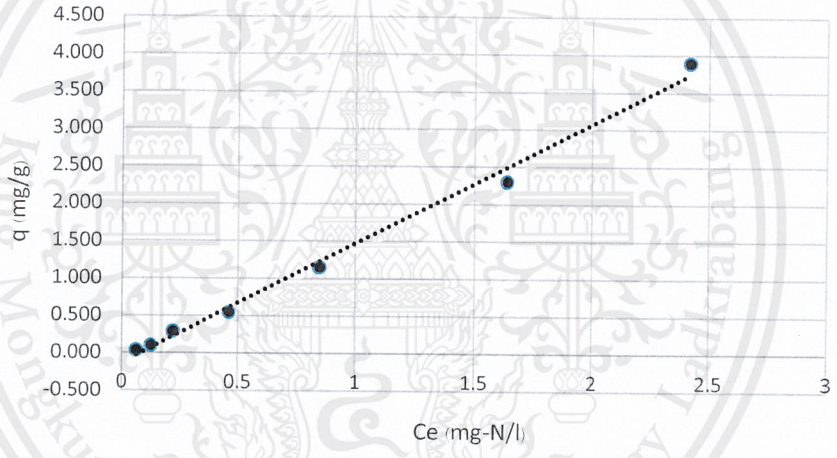


Figure 4.5. Adsorption isotherm of ammonia nitrogen using zeolite A adsorbent.

The equilibrium experimental data for the adsorption of ammonia nitrogen from aqueous onto zeolite A adsorbent was compared using Langmuir and Freundlich models as shown in Figures 4.4 and 4.5 respectively.

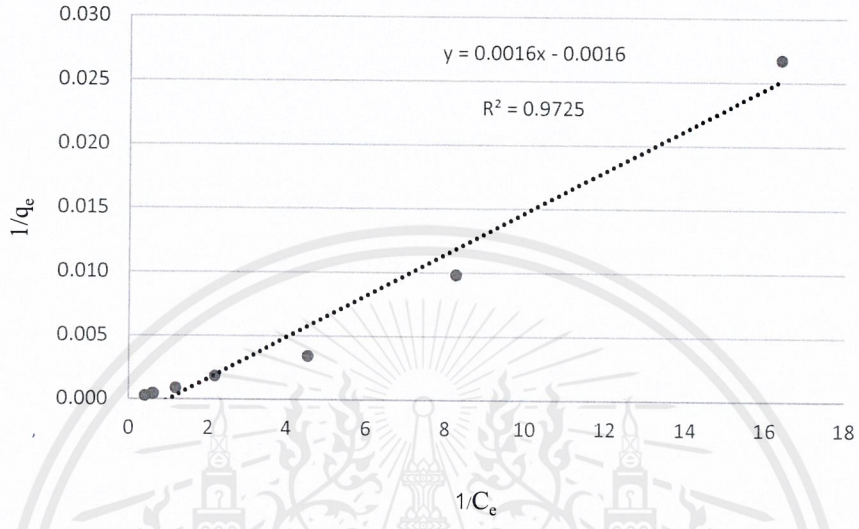


Figure 4.6. Langmuir plots for adsorption of ammonia nitrogen onto zeolite A adsorbent.

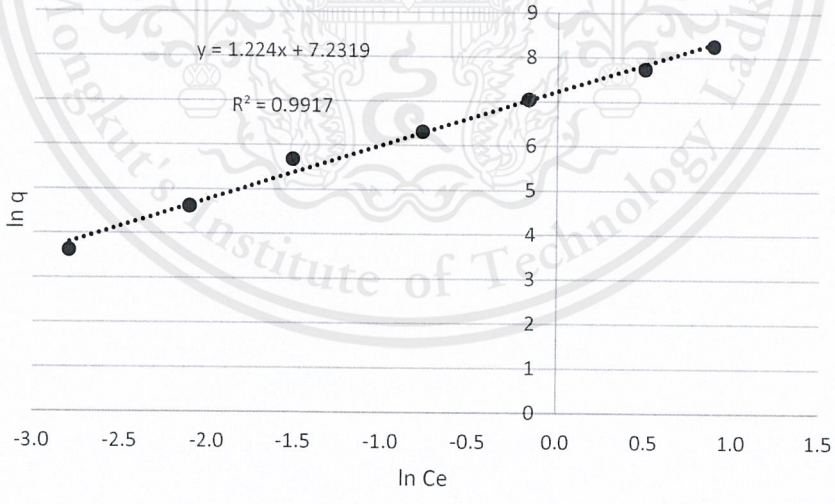


Figure 4.7. Freundlich plots for adsorption of ammonia nitrogen onto zeolite A adsorbent

As shown in figure 4.4 and 4.5, the obtained equation of Langmuir and Freundlish adsorption model is $qe = \frac{0.625Ceq}{1+0.16Ceq}$, $R^2 = 0.9723$ and $qe = 1.978Ce^{1.223}$, $R^2 = 0.9917$ respectively. Isotherm parameters were calculated from graph and presented in Table 4.1. Therefore, the Freundlich model gives the best correlation coefficient value for ammonia nitrogen adsorption isotherm using zeolite A adsorbent based on the obtained determination coefficient, $R^2 = 0.9917$. It is evident from these data that the adsorption sites occurring between zeolite A and ammonia nitrogen molecules are multilayer adsorption. Charge on the surface of zeolite A is a determining factor of ammonia nitrogen removal efficiency. Ammonium ion (NH_4^+) which has a positive charge will attract negative charge on the surface of zeolite A adsorbent [20].

Table 4.1. Langmuir and Freundlich parameters for the adsorption of ammonia nitrogen onto zeolite A

Langmuir Isotherm			**Freundlich Isotherm**		
q_{max} (mg/g)	K_L (L mol^{-1})	R^2_L	K_F (L g^{-1})	n	R^2_F
0.625	1	0.9723	1.978	0.817	0.9917

CHAPTER V
CONCLUSION

5.1 Conclusion

The presented study was carried out to investigate the adsorption capacity of rice straw, delignified rice straw, and zeolite A as alternative adsorbents for ammonia nitrogen removal. The experimental results clearly indicated that zeolite A was an effective adsorbent for the removal of ammonia nitrogen from aqueous solution. However, rice straw and delignified rice straw were not able to provide the adsorption capacity of ammonia nitrogen. The influence of ammonia nitrogen concentration had significant effect on the removal capacity of zeolite A. The rate of ammonia nitrogen removal was rapid after adsorption processes conducted due to the high surface area of adsorption sites and the capacity to enhance the cation exchange with NH_4^+ from ammonia solution. For the application of Langmuir and Freundlich equations, the experimental results showed that the Freundlich model gives the best correlation coefficient value based on the obtained determination coefficients (R^2) of Freundlich model gives the highest value. Therefore, it employed to describe the adsorption sites between zeolite A and ammonia nitrogen are multilayer adsorption. Zeolite A provides the application potentials for effective ammonia nitrogen removal and may display the advantages of adsorption capacity for other hazardous pollutants from aqueous solution.

5.2 Suggestions

1. The adsorption study should be further carried out with regular wastewaters from industrial or urban uses to investigate the actual adsorption capacity of determined adsorbent.
2. Zeolite A adsorbent should be further studied to adjust for the higher adsorption capacity and wider adsorption applications.

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APPENDIX

APPENDIX A

STANDARD CURVE

APPENDIX A

STANDARD CURVE

Standard curve of ammonia nitrogen solution was prepared at 0.125, 0.25, 0.5, 1.0, 2.0, 4.0, and 8.0 mg-N/l respectively. The absorbance values of each ammonia nitrogen concentration were recorded at 640 nm in visible spectrophotometer with a 10 cm length cuvette. The relation between ammonia nitrogen concentrations and absorbance values are presented in figure A.1. The correlation equation is $y = 0.2792x + 0.0153$, $R^2 = 0.9993$.

Table A.1. Experimental data of ammonia nitrogen concentration (mg-N/l) and absorbance value

Concentration of ammonia nitrogen (mg-N/l)	Absorbance
0	0
0.125	0.043
0.25	0.088
0.5	0.170
1	0.303
4	1.128

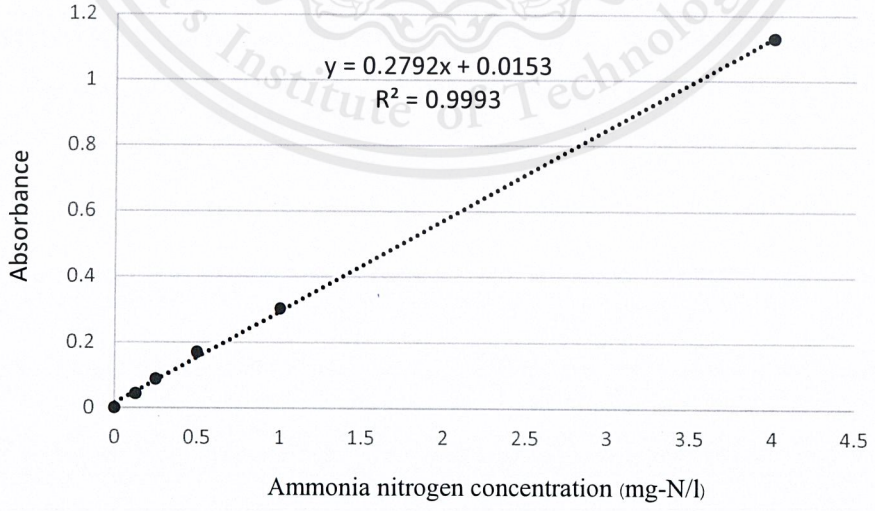

Figure A.1. The correlation curve between ammonia nitrogen concentration (mg-N/l) and absorbance value.

APPENDIX B

EXPERIMENTAL DATA

APPENDIX B

EXPERIMENTAL DATA

Table B.1. The experimental data of ammonia nitrogen adsorption onto 1 g of zeolite A adsorbent, 0.25 mg-N/l initial ammonia nitrogen concentration

time (min)	Ammonia nitrogen concentration (mg-N/l)	% removal	q (mg/g)
0	0.217	0	0
1	0.122	42.412	0.094
3	0.118	44.194	0.098
5	0.122	42.417	0.094
10	0.122	42.419	0.094
15	0.122	42.414	0.094
20	0.118	44.190	0.098
30	0.118	44.193	0.098
60	0.118	44.195	0.098
90	0.115	45.987	0.102
120	0.115	45.982	0.102

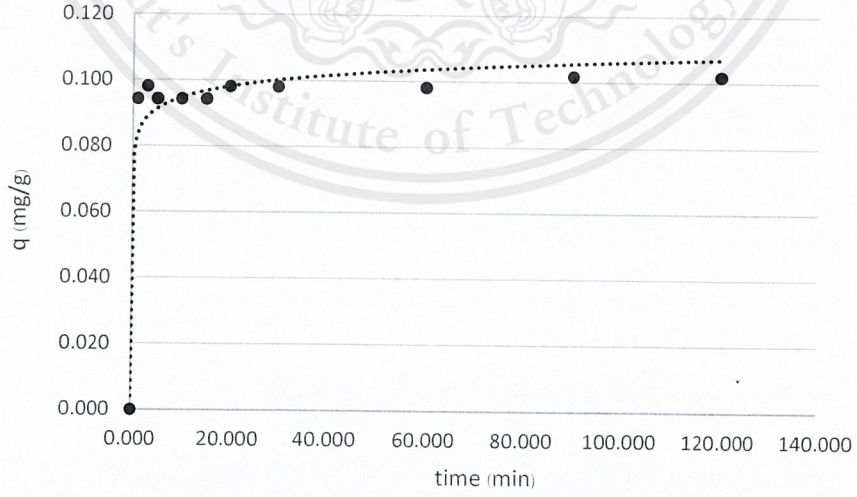

Figure B.1. The correlation curve of the amounts of ammonia nitrogen adsorbed onto unit mass of zeolite A (mg/g) with time, 0.25 mg-N/l initial ammonia nitrogen concentration.

Table B.2. The experimental data of ammonia nitrogen adsorption onto 1 g of zeolite A adsorbent, 0.5 mg-N/l initial ammonia nitrogen concentration

time (min)	Ammonia nitrogen concentration (mg-N/l)	% removal	q (mg/g)
0	0.513	0	0
1	0.242	52.933	0.272
3	0.260	49.356	0.253
5	0.220	57.225	0.294
10	0.238	53.648	0.275
15	0.227	55.794	0.286
20	0.245	52.217	0.268
30	0.220	57.225	0.294
60	0.227	55.794	0.286
90	0.220	57.225	0.294
120	0.220	57.225	0.294

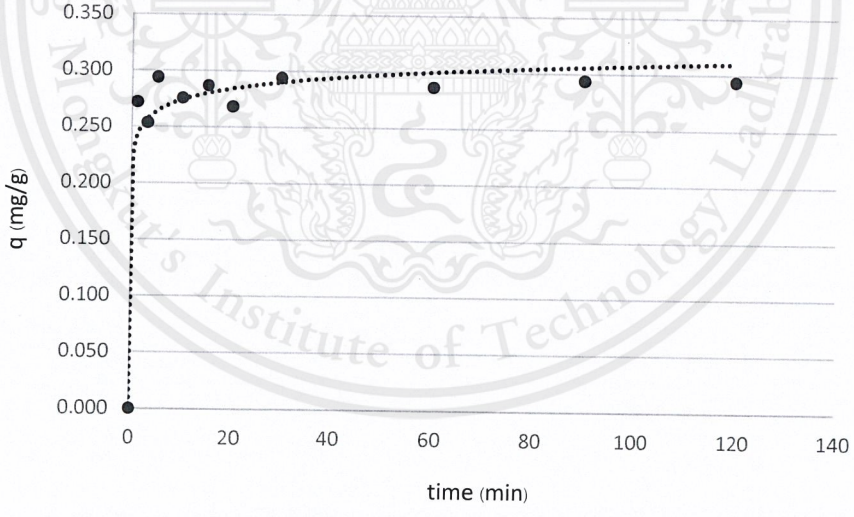

Figure B.2. The correlation curve of the amounts of ammonia nitrogen adsorbed onto unit mass of zeolite A (mg/g) with time, 0.5 mg-N/l initial ammonia nitrogen concentration.

Table B.3. The experimental data of ammonia nitrogen adsorption onto 1 g of zeolite A adsorbent, 1.0 mg-N/l initial ammonia nitrogen concentration

time (min)	Ammonia nitrogen concentration (mg-N/l)	% removal	q (mg/g)
0	1.013	0	0
1	0.469	53.662	0.544
3	0.462	54.387	0.551
5	0.484	52.212	0.529
10	0.466	54.025	0.547
15	0.466	54.025	0.547
20	0.491	51.487	0.521
30	0.477	52.937	0.536
60	0.477	52.937	0.536
90	0.473	53.299	0.540
120	0.462	54.387	0.551

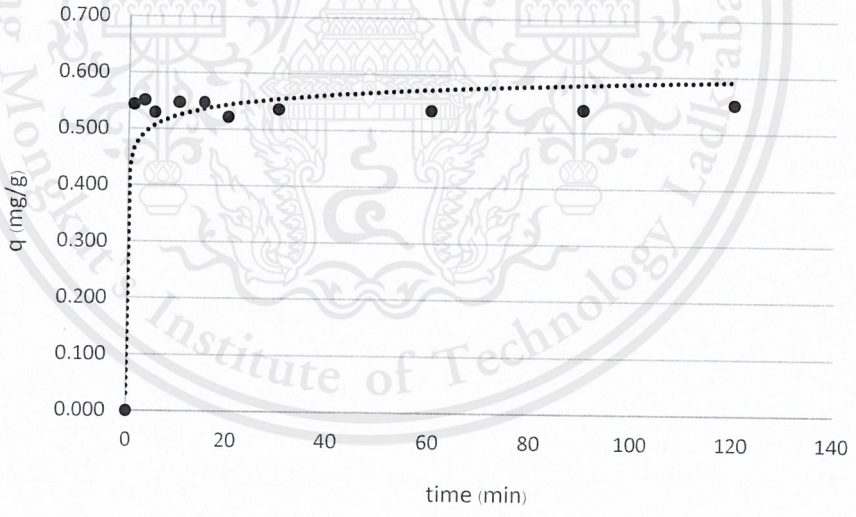

Figure B.3. The correlation curve of the amounts of ammonia nitrogen adsorbed onto unit mass of zeolite A (mg/g) with time, 1.0 mg-N/l initial ammonia nitrogen concentration absorbance values.

Table B.4. The experimental data of ammonia nitrogen adsorption onto 1 g of zeolite A adsorbent, 2.0 mg-N/l initial ammonia nitrogen concentration

time (min)	Ammonia nitrogen concentration (mg-N/l)	% removal	q (mg/g)
0	2.001	0	0.
1	0.939	53.047	1.061
3	0.855	57.269	1.146
5	0.848	57.636	1.153
10	0.859	57.085	1.142
15	0.870	56.535	1.131
20	0.848	57.636	1.153
30	0.851	57.452	1.149
60	0.870	56.535	1.131
90	0.851	57.452	1.149
120	0.848	57.636	1.153

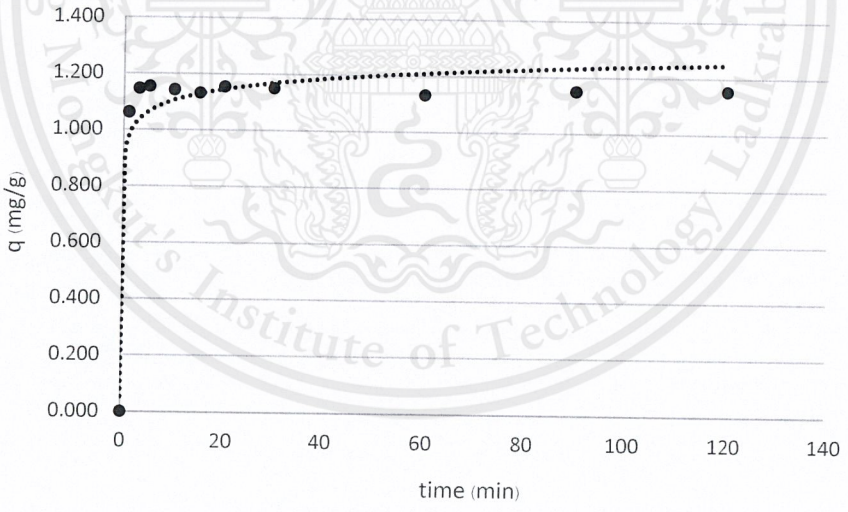

Figure B.4. The correlation curve of the amounts of ammonia nitrogen adsorbed onto unit mass of zeolite A (mg/g) with time, 2.0 mg-N/l initial ammonia nitrogen concentration.

Table B.5. The experimental data of ammonia nitrogen adsorption onto 1 g of zeolite A adsorbent, 4.0 mg-N/l initial ammonia nitrogen concentration

time (min)	Ammonia nitrogen concentration (mg-N/l)	% removal	q (mg/g)
0	3.942	0	0
1	1.639	58.417	2.303
3	1.668	57.691	2.274
5	1.675	57.509	2.267
10	1.654	58.054	2.289
15	1.657	57.963	2.285
20	1.668	57.691	2.274
30	1.693	57.055	2.249
60	1.632	58.599	2.310
90	1.643	58.327	2.299
120	1.639	58.417	2.303

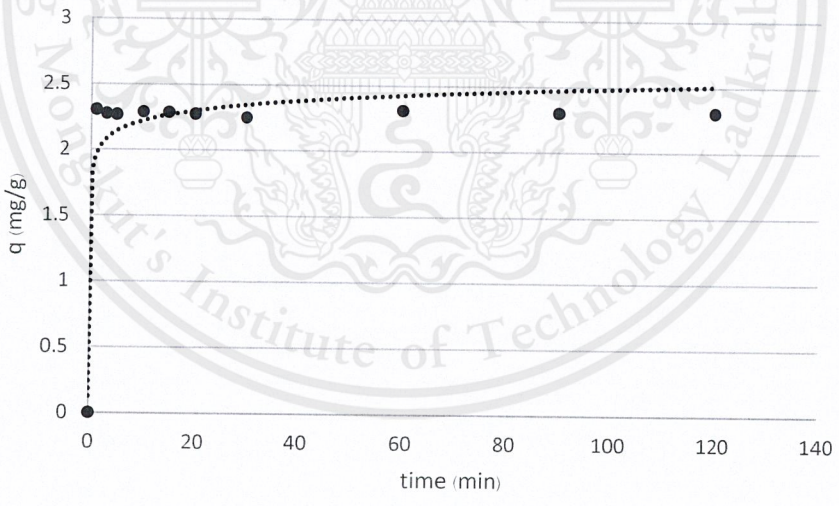

Figure B.5. The correlation curve of the amounts of ammonia nitrogen adsorbed onto unit mass of zeolite A (mg/g) with time, 4.0 mg-N/l initial ammonia nitrogen concentration.

Table B.6. The experimental data of ammonia nitrogen adsorption onto 1 g of zeolite A adsorbent, 8.0 mg-N/l initial ammonia nitrogen concentration

time (min)	Ammonia nitrogen concentration (mg-N/l)	% removal	q (mg/g)
0	6.316	0	0
1	2.605	58.750	3.711
3	2.655	57.956	3.660
5	2.419	61.699	3.897
10	2.433	61.472	3.883
15	2.390	62.153	3.926
20	2.512	60.225	3.804
30	2.419	61.699	3.897
60	2.340	62.947	3.976
90	2.319	63.287	3.997
120	2.419	61.699	3.897

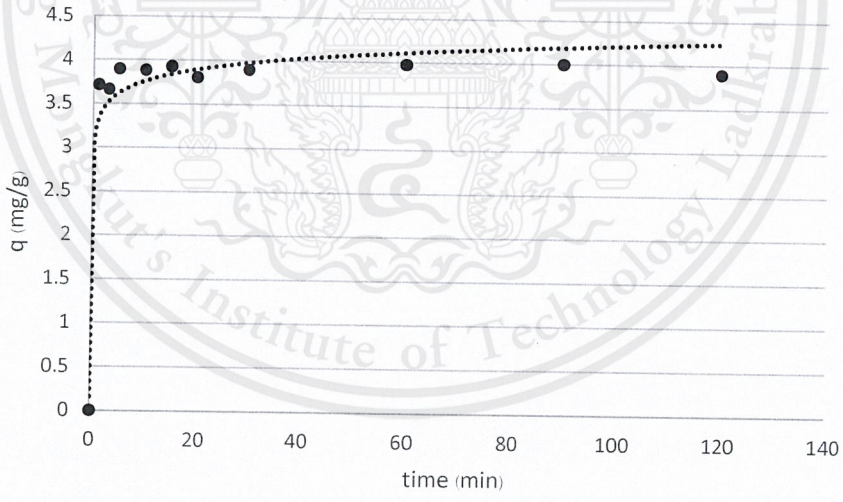

Figure B.6. The correlation curve of the amounts of ammonia nitrogen adsorbed onto unit mass of zeolite A (mg/g) with time, 8.0 mg-N/l initial ammonia nitrogen concentration.

Table B.7. The experimental data of ammonia nitrogen adsorption onto 1 g of rice straw adsorbent, 0.5 mg-N/l initial ammonia nitrogen concentration

time (min)	Ammonia nitrogen concentration (mg-N/l)	% removal	q (mg/g)
0	0.501	0	0
1	0.481	3.992	0.020
3	0.494	1.397	0.007
5	0.496	0.998	0.005
10	0.498	0.598	0.003
15	0.495	1.197	0.006
20	0.493	1.596	0.008
30	0.499	0.399	0.002
60	0.496	0.998	0.005
90	0.496	0.998	0.005
120	0.494	1.397	0.007

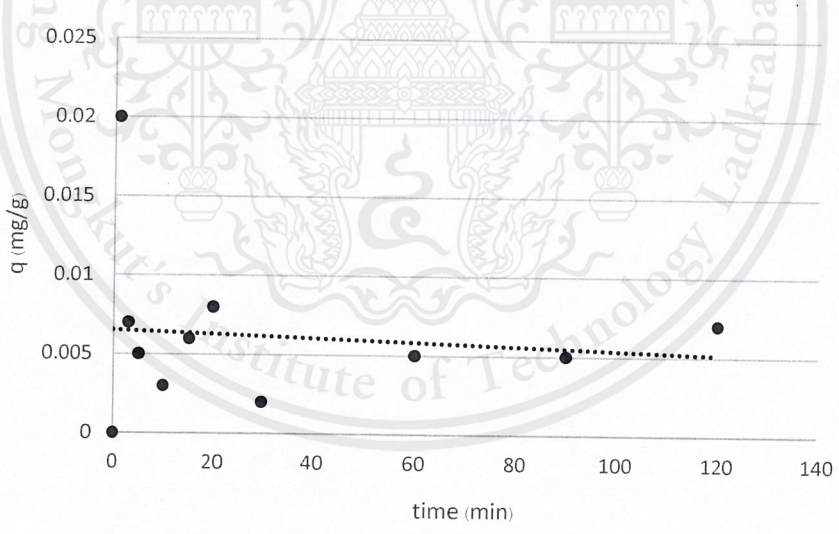

Figure B.7. The correlation curve of the amounts of ammonia nitrogen adsorbed onto unit mass of rice straw (mg/g) with time, 0.5 mg-N/l initial ammonia nitrogen concentration.

Table B.8. The experimental data of ammonia nitrogen adsorption onto 1 g of delignified rice straw adsorbent, 0.5 mg-N/l initial ammonia nitrogen concentration

time (min)	Ammonia nitrogen concentration (mg-N/l)	% removal	q (mg/g)
0	0.489	0	0
1	0.492	1.796	0.009
3	0.492	1.796	0.009
5	0.467	6.786	0.034
10	0.490	2.195	0.011
15	0.496	0.998	0.005
20	0.49	2.195	0.011
30	0.495	1.197	0.006
60	0.492	1.796	0.009
90	0.489	2.395	0.012
120	0.494	1.397	0.007

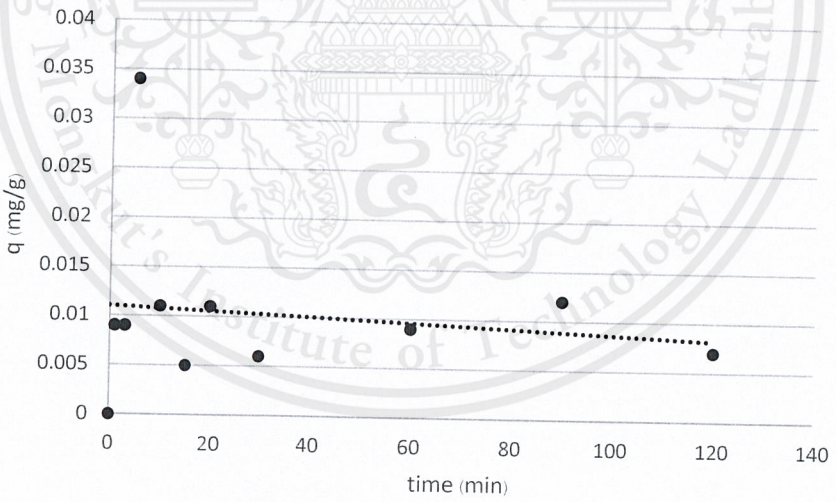

Figure B.8. The correlation curve of the amounts of ammonia nitrogen adsorbed onto unit mass of delignified rice straw (mg/g) with time, 0.5 mg-N/l initial ammonia nitrogen concentration.

APPENDIX C

PICTURES

APPENDIX C

PICTURES

Figure C.1. Collected rice straw

Figure C.2. Differences of ammonia nitrogen concentration

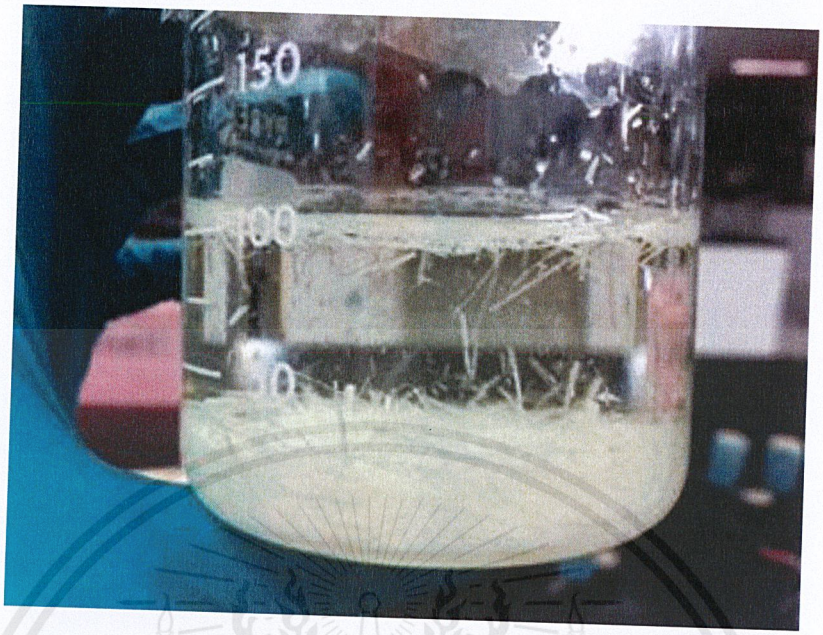

Figure C.3. Delignified rice straw

Figure C.4. Different physical characteristic of rice straw and delignified rice straw

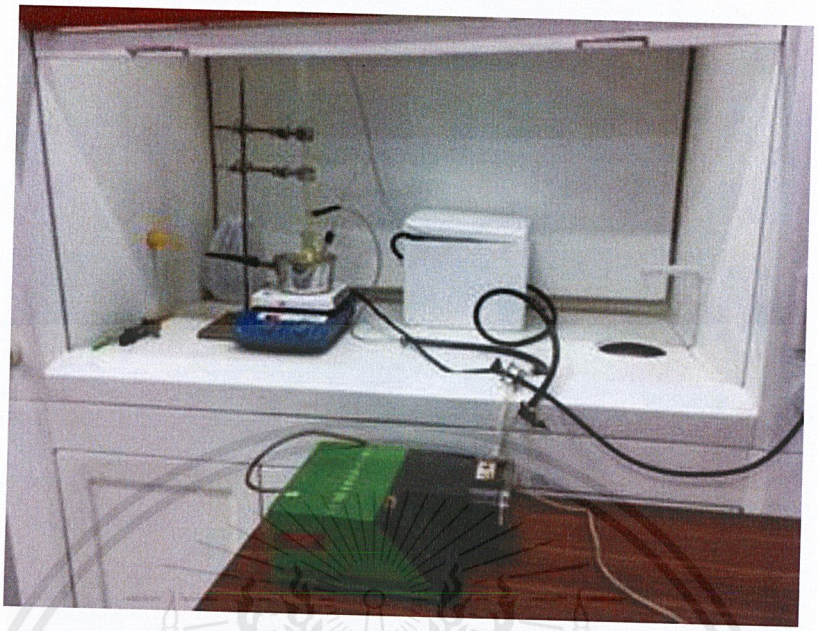

Figure C.5. Reflux condenser setup for the delignification of rice straw and zeolite A synthesis

Figure C.6. Obtained zeolite A

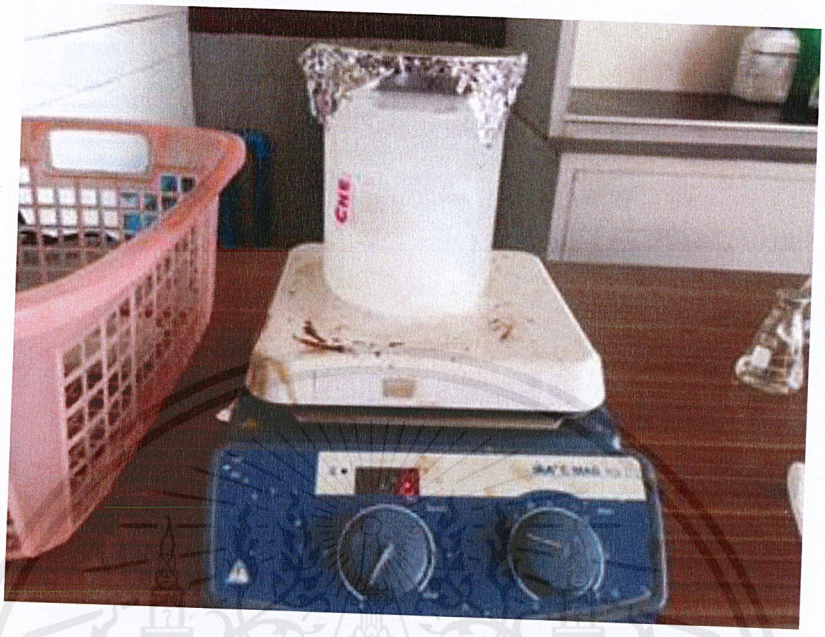

Figure C.7. Ammonia nitrogen adsorption using zeolite A adsorbent