Journal of Advanced Research in Applied Sciences and Engineering Technology

Journal homepage:
https://semarakilmu.com.my/journals/index.php/applied_sciences_eng_tech/index
ISSN: 2462-1943

# Adsorption of Sodium Ion ($Na^+$) onto Synthesized Zeolite-A from Malaysian Kaolin by Hydrothermal Method: Equilibrium and Kinetic Studies

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**ARTICLE INFO**

***Article history:***
Received 10 January 2023
Received in revised form 18 May 2023
Accepted 25 May 2023
Available online 13 June 2023

***Keywords:***
Adsorption; adsorption isotherm; kaolin; kinetic; zeolite

**ABSTRACT**

Batch adsorption of sodium ion ($Na^+$) using Malaysian kaolin-based materials, zeolite A was studied and observed. The experiments were done at different parameters such as varying initial concentrations of sodium ions in seawater solution (70 to 280 mg $L^{-1}$), pH range of 6 to 8 and various adsorbent dosages (0.1 to 0.4g). The Langmuir and Freundlich's isotherms were used to analyse the data, and the results were compared. This study discovered that the Langmuir isotherm matched the experimental data very well, with the maximum monolayer adsorption capacities derived from the Langmuir equation being 769.3 mg $g^{-1}$. Using pseudo-first-order, pseudo-second order, and intraparticle diffusion models, the kinetics of the reaction were further investigated and understood. The Intra Particle Diffusion Kinetic Equation was the most accurate description of the adsorption kinetics of sodium ions onto zeolite synthesis.

## 1. Introduction

Seawater is one of the water bodies that cover most parts of the world. Brackish water, seawater, and wastewater are three desalination sources [1]. Desalination is when salt and other mineral products are removed from water [2]. This method is tailored for a specific function such as manufacturing, drinking, agriculture and lastly, aquaculture [1]. Multiple desalination methods are used, including evaporation, distillation, ion exchange, adsorption, freezing, reverse osmosis and electro dialysis [2]. Desalination technologies are available to remove salts to produce drinkable water from seawater. With regards to the increasing need for fresh and clean water globally, an improved and economical approach using ion exchange and desalination is, therefore, more desirable. In comparison, the adsorption method was commonly used and successfully applied with

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https://doi.org/10.37934/araset.31.1.5367

various forms of adsorbents, such as zeolite, activated carbon, alumina, and silica gel, in separation industries decades ago [3].

Adsorption can really be defined as the consumption of liquid molecules from a mixture onto the surface of a solid. The basic components of one step of the fluid are moved onto the solid adsorbent surface via a separation process [4]. Adsorption provides a simple, low-cost method with low energy consumption [5]. Adsorption is one of the ways that salts and minerals can be eliminated from seawater. To date, the adsorption approach has been greatly employed since it is a simple, high-efficiency removal method [6,7]. Adsorption is a method for extracting a wide range of substances from wastewater, mainly from industrial sources [8]. Adsorption is also frequently used to remove low levels of non-biodegradable organic pollutants from aquifers and to prepare water supply. When liquid molecules bind to the surface of a solid material, this is known as adsorption [9]. An insoluble adsorbent material with a porous structure allows liquid to easily coat its surface, including capillaries and pores [10].

Many studies have shown that adopting zeolites for water desalination may reduce contaminants by up to 99.99 per cent using a number of zeolite columns, depending on the zeolite type and salt concentration [11,12]. Since seawater is high in sodium ions, it is recommended that a Na-free zeolite be used to extract sodium ions. The desalination technique is an innovative application of zeolite membranes for hazardous waste treatment, but various inorganic membranes have been widely employed in reverse osmosis and membrane distillation procedures to remove radioactive liquids [13-16]. Zeolites are a class of microporous aluminosilicate. The zeolite frame is an extremely complicated three-dimensional (3D) structure with cation adsorption and replaceable characteristics [17]. In fact, the framework of zeolite is composed of silicon and aluminium atoms. A three-dimensional crystal featuring cavities in multiple directions allows zeolites for ion exchange [18]. The pore structure, secondary building unit, and frame density all play a role in categorizing zeolite formations [19]. Zeolite contains around 20 to 50 per cent of the void's volume. It has an internal surface area of one hundred thousand square meters per kilogram, allowing zeolite to be a unique adsorbent [20]. There are many factors influencing the efficiency of zeolite adsorption, including its pore size, exchangeable ions, and the zeolite's chemical composition.

By emulating the formation of natural zeolite in the 1940s, the hydrothermal process successfully synthesized zeolite from natural or synthetic resources, resulting in high demand for zeolite production for industrial purposes. The need for industries for high-uniformity pores and high zeolite ion exchangeability makes the demand for synthetic zeolites higher than natural zeolites. However, given the cost disadvantage of synthetic zeolites, when only small quantities of zeolites are required, natural zeolites are chosen. Synthetic zeolites may be made for specific purposes, such as the process of adsorption [21]. To improve hydrothermal synthesis, a more sophisticated treatment method involving alkaline fusion, zeolite synthesis in the molten state and microwave-assisted zeolite formation was used [22,23].

To The suitability of synthesized zeolite-A from kaolin in this work has been assessed for sodium-ion in seawater solution adsorption. The influences of the initial sodium ion concentration, dose, and duration were investigated, as well as the isotherm and kinetic data.

## 2. Methodology

### *2.1 Isotherm Modelling*

Considering a relatively uniform surface with a small number of identical sites and little interaction among adsorbate, Eq. (1) yields the Langmuir isotherm that is appropriate for monolayer sorption [24,25].

$$qe = \frac{q_m K_L C_e}{1+K_L C_e} \tag{1}$$

The amounts $q_e$ (mg $g^{-1}$) and Ce (mg $L^{-1}$) denote the quantities of sodium adsorbate concentration per unit mass of adsorbent and the amounts of adsorbed sodium ion concentration in solution at equilibrium, respectively. The $q_m$ is the maximum amount of adsorbate per unit mass of adsorbent required to form a complete monolayer on the surface-bound at high Ce (mg $L^{-1}$), and $K_L$ is a constant that is associated with the affinity of the binding sites. Eq. (2) gives the linear form of the Langmuir isotherm equation

$$\frac{1}{q_e} = \left(\frac{1}{K_L\, q_m}\right)\left(\frac{1}{C_e}\right) + \frac{1}{q_m} \tag{2}$$

Introducing the dimensionless equilibrium factor, Eq. (3) can be used to describe the Langmuir isotherm

$$R_L = \frac{1}{[1+bC_0]} \tag{3}$$

$R_L$ =1 linear; $R_L$ =0 reversible; $R_L$ >1 unfavourable; 0< $R_L$ <1 favourable [26]. This isotherm model is derived from empirical evidence. In order to investigate multilayer adsorption with a non-uniform dispersion of adsorption heat over a heterogeneous surface, this model can be used [27,28]. Eq. (4) expresses the Freundlich isotherm in its linear form

$$log\ q_e = log\ k_f + \left(\frac{1}{n}\right) log\ C_e \tag{4}$$

where $q_e$ is the solute liquid phase equilibrium concentration in mg/g, Ce is the sorbate liquid phase equilibrium concentration in mg/L, $K_f$ is the Freundlich constant, the dimensionless constant, n. The constant 'n' represents the deviation from the linearity of adsorption (Table 1).

**Table 1**
The constant 'n' from linearity of adsorption

| n value | Significance |
|---|---|
| n=1 | Linear |
| n<1 | Chemical Sorption Operation |
| n>1 | Good |

### *2.2 Kinetic Adsorption*

The study of adsorption kinetics can provide a detailed description of the rate at which solubility occurs. This rate can be used to monitor the residual lifetime of solid surface and solution material [29]. This parameter is essential for the modelling and evaluation of the adsorption process to enhance the treatment process's efficiency [30]. To understand the adsorption mechanism of pollutant material in a solution that occurs on the adsorbent, information about the kinetic model is so important [31]. The explanation of which differences in concentration of the two substances occur is the solution reaction, where dissolved solids react with an adsorbent. The first pseudo-order is a reaction rate of the first order used for the phase, which can be expressed by Eq. (5) [32]

$$d_{qt} = K_1 [q_e - q_t] \tag{5}$$

The equation can be rewritten in a straight-line equation as follow

$$\ln[q_e - q_t] = \ln q_e - [K_1][t] \tag{6}$$

$q_e$ (mg/g): equilibrium-state adsorption capacity,
$q_t$ in (mg/g): adsorption capacity at time t,
$K_1$ ($min^{-1}$): constant

Each $q_e$ and $K_1$ value can be determined from y-intercept and gradient graph ln ($q_e$ - $q_t$) against t, adsorption time. Despite that, the first pseudo-order equation is not very well-fitted over the entire range of contact time [29]. So, the second pseudo-order was introduced for occurrences of chemical occurrences, and then the reaction occurs at the adsorbent surface. The pseudo second order equation, as in Eq. (7)

$$\frac{d_{qt}}{d_t} = [K_2][q_{e-}q_t]^2 \tag{7}$$

where $K_2$ is the constant rate for the second pseudo-order adsorption reaction. The above equation is changed to form an equation of linearity to be like the following Eq. (8)

$$\frac{1}{q_t} = \left[\frac{1}{K_2 q_e^2}\right]\left[\frac{1}{t}\right] + \frac{1}{q_e} \tag{8}$$

Because the adsorption process, which occurs on a porous adsorbent, is a multi-step process, the intra-particle model can be explained successfully according to Halim *et al.,* [33] and Igwe *et al.,* [34].

### *2.3 Intra Particle Diffusion*

The whole process of adsorption may be controlled by either one or more steps or a combination of more than one step, as presented by Weber *et al.,* [31] in Eq. (9)

$$q_t = K_i t_{0.5} + C \tag{9}$$

The value of c is a constant, while ki is the intra-particle diffusion constant rate (mg/g $min^{0.5}$). The values of c and ki are respectively determined from the y-axis intercept and gradient graph $q_t$ against adsorption time, $t_{0.5}$. The constant t represents the contact time in (minutes). C is the film thickness, and the greater the value of C obtained means that the effect of the fluid film on the adsorption process is high.

## **3. Methodology**

### *3.1 Preparation of Adsorbent*

Because the kaolin condition has a greater lumpy size, a pre-treatment technique is required before synthesis can commence. The kaolin lumps must be crushed and ground before they can be used. It was decided that the lump-sized kaolin would be crushed using the ball milling machine. A 15L bucket was filled with distilled water and 3 kg of grounded kaolin and left to rest for a week. The kaolin that has been soaked is stirred often. To remove all the solid particles during decanting, the kaolin had to be stirred, the floating dirt was decanted, and the supernatant was decanted. The container's bottom has been cleared of any remaining solids due to this operation. After leaving at room temperature for 24 hours, the fine particle was dried and sieved using a 63-micron filter. Then, it was dried in an oven for 12 hours before being heated in the furnace for four hours at a temperature of 650°C for the metakaolin process. Slowly, 3 g of metakaolin was added to the 60 mL diluted NaOH solution. The composition of the prepared reaction mix was based upon Chandrasekhar and Chandrasekhar *et al.,* [35] work. For the ageing procedure, the mixture was kept at 40°C for 24 hours. Evaporation was prevented by blending the material in an enclosed area. During the ageing process, the crystallization procedure is carried out in a 100mL Teflon line autoclave with a continuous solution transfer. It ran for 12 hours at 100°C. After that, a cold-water tray is used to quickly chill the mixture. Following the procedure's conclusion, the supernatant was discarded, and the pH was dropped to less than 10. After 12 hours in the oven at 60°C, the final solid was dried. To make sure zeolite A was obtained, the final solid was examined for physical and chemical characteristics.

### *3.2 Preparation of Adsorbate*

The five litres of seawater sample were taken from the beach. The seawater sample was straightforwardly move to the laboratory. Seawater pH is ranged from 6 to 8. The seawater was diluted with distilled water to produce 70-280 mg $L^{-1}$ sodium ion concentration.

### *3.3 Primarily Batch Adsorption Study*

#### *3.3.1 Effect of various volumes of seawater solution*

The main purpose of a batch experiment was to determine the most suitable condition for eliminating sodium ions in seawater solution by using Zeolite-A. The parameters used in the experiment were amount of adsorbent, time, and initial sodium ion concentration. The pre-experiment started with different volumes of seawater solution (50, 75, 100 and 150 ml) at a constant mass of zeolite A (0.1g) and 280 mg/L of initial sodium ion concentration and with 180 minutes

#### *3.3.2 Effect of various sodium ion concentration*

The effect of adsorbate removal was studied by varying the initial sodium ion concentrations in the solution (70, 140 and 280 mg/L and a constant 100 ml seawater solution, 0.1 g zeolite A and 240-minute contact time).

#### *3.3.3 Isotherm study*

The adsorbent mass has a strong effect on the process of adsorption [9]. The experiment was designed to study the effect of different dosages of zeolite A (0.1, 0.2, 0.3 and 0.4 g) against sodium ion adsorption in seawater solution with constant initial sodium ion concentration, 290 mg/L, time (240 minutes) and volume (100 ml). The optimum parameter taken in the previous experiment allowed the study of isothermic adsorption. Various zeolite A dosages are used in performing the isothermic study in relation to time and other constant parameters that are volume solutions. A set of 100ml seawater solution beakers is prepared for the varying mass of Zeolite-A. After shaking for

10 minutes, the sample was collected at a uniform interval. The sample was analysed using a sodium ion detector (HI98190 +FC300, combination sodium electrode) by Hanna Instrument.

### *3.3.4 Kinetic study*

For the kinetic study, the other parameter, initial concentration of sodium ions (280 mg/L), volume (100ml), zeolite mass (0.1 g) and pH 6 to 8, are constant with respect to the various time interval. All of the experiments have been repeated three times for the exact result.

## 4. Results

### *4.1 Effect of Various Volume Seawater Solution*

Figure 1 shows the differences in the removal of sodium ion ($Na^+$) in seawater due to the volume/mass change proportion using a different volume of seawater solution (50, 75, 100 and 150 ml) at a constant density of 0.1 g zeolite-A powder. It is clearly seen that the rate of removal of sodium ions increased sharply with the volume of seawater solution increasing from 50 to 100ml. However, the sodium-ion removal rate is approximately the same at the seawater volume of 100 to 150ml solution. It is due to the explained agglomeration of the active solid stage in which the removal rate of sodium-ion at 50 ml solution is low. Thus, the 100ml volume was chosen for the upcoming experiment.

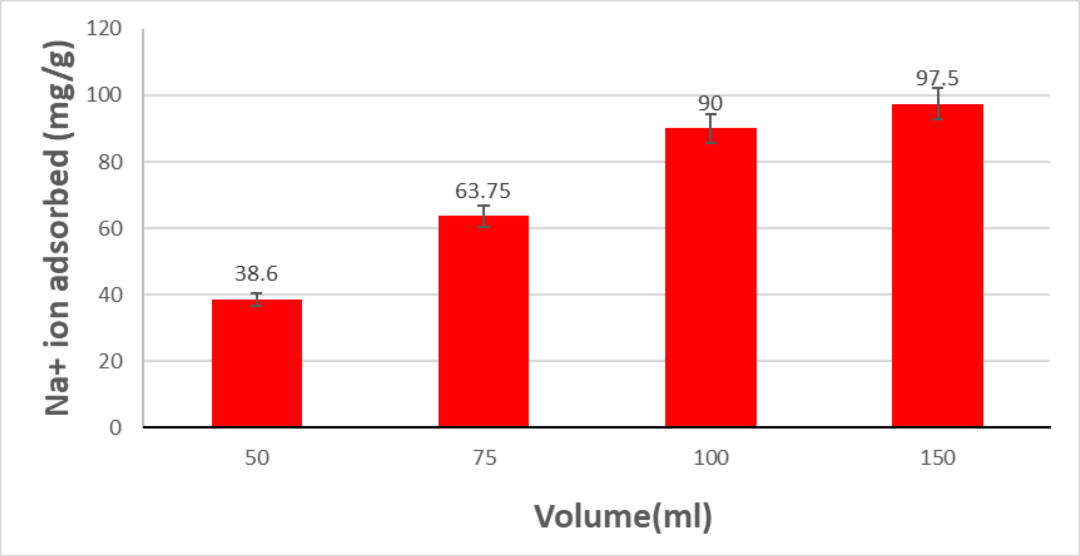

**Fig. 1.** Volume/mass proportion of sodium ion adsorption

### *4.2 Effect of Sodium Ion Concentration*

The adsorption capacity increased over time graph was plotted in Figure 2. In any case, the decrease in the response of the main force combined with the reduction of the dynamic site makes the procedure takes quite a while before achieving equilibrium [36]. The result shows the time required for all experiments to achieve equilibrium in the adsorption process is around 240 minutes. So, for the next experiment, 240 minutes were chosen.

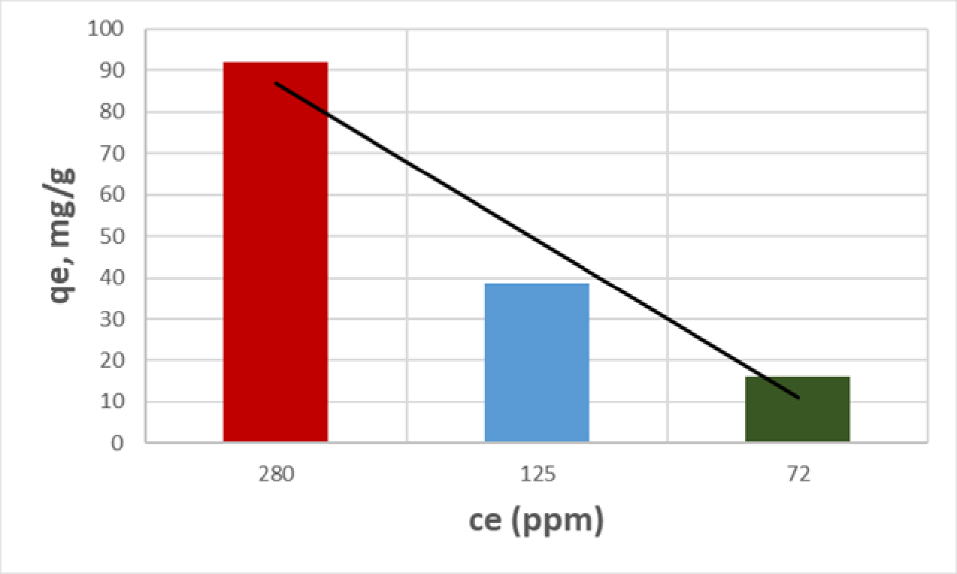

**Fig. 2.** Different of sodium ion concentration at 240 minutes

### *4.3 Effect of Dosage*

Figure 3 is the amount of sodium-ion removal from seawater with respect to the different dosages of adsorbents used, with the constant of other parameters, which is the time (240 minutes) and volume (mL/g), the sodium-ion adsorption efficiency shows the increment of the adsorbent dose (0.1 to 0.4 g/L). The most extreme removal was seen with a dosage of 0.40 mg/L adsorbent. The adsorption capacity, $q_e$, decreases with an increased dosage of adsorbents. Increasing the adsorbent dose has made the availability of adsorption area increase, and it becomes the reason for the adsorption site to remain unsaturated since the adsorption capacity decreases when the adsorbent dosage is increased [37,38].

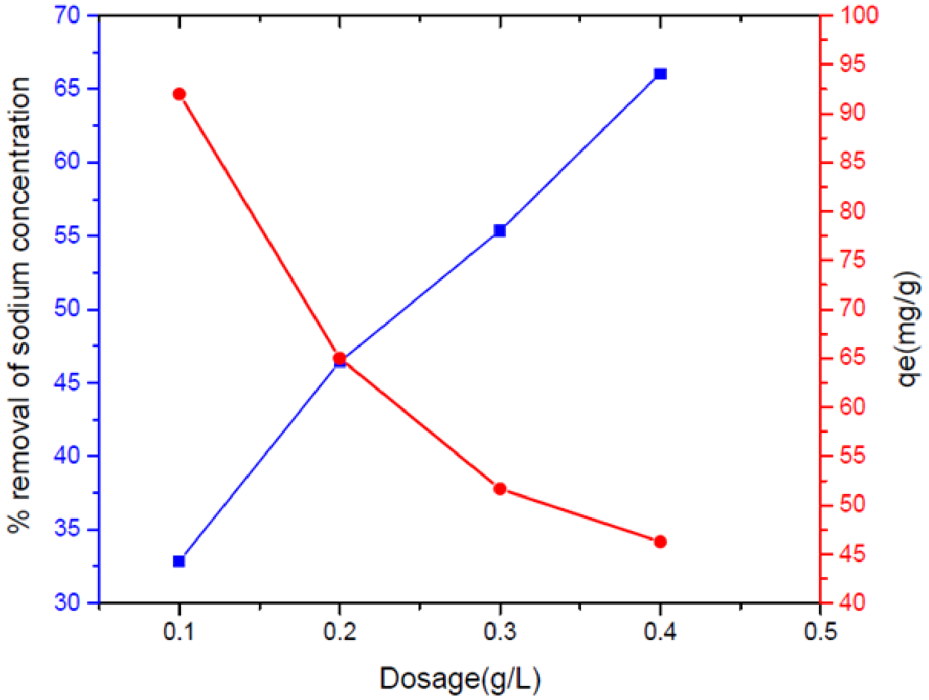

**Fig. 3.** Different of adsorbent dosage

### *4.4 Adsorption Isotherm*

Figure 4 represent the correlation diagram of adsorption isotherm of Langmuir for sodium adsorbate on Zeolite-A, whereas Figure 5 depicts Freundlich's linear graph isotherm for $Na^+$ adsorption to Zeolite-A. Furthermore, the correlation coefficient ($R^2$), adsorption constant and capacity of adsorption for adsorption of sodium ion to zeolite is shown in Table 2. There is no significant difference in the adsorption, $R^2$, from the linear plot for both Langmuir and Freundlich. This difference in correlation will determine the accuracy of the experiment data to the isotherm model [39]. From the Langmuir isotherm model, the value of $R^2$=0.9074, while for Freundlich, the value of $R^2$ is 0.9028. Despite that there is no notable difference between the two isotherms, the Langmuir model was chosen as the best model for adsorption done in this research. Some researchers fit the research to Langmuir compared to Freundlich and vice versa. Langmuir's model shows the finest model to fit the removal of ammonium using zeolite [40]. However, Du *et al.,* [41] and Widiastuti *et al.,* [42] presented different results where the authors claimed that their experimental data are best fitted by the Freundlich model.

Further studies indicate that such differences must have occurred due to the nature of zeolite used in their studies. The adsorption capacity, $q_m$ is the size of the maximum saturated molecule adsorption capacity with full coverage of a single layer [42]. The maximum adsorption capacity for sodium-ion in seawater solution, as chosen in Table 2, was found to be 769.3 mg/g. Using the Langmuir isotherm on the experimental data suggested that the chemical adsorption mechanism is applicable at the beginning of the process (chemisorption). It also indicates the potential for a single layer of adsorption to have occurred on the adsorbent surface. This is in line with the assumption that the Langmuir isotherm that the sorption takes place within the adsorbent at a given site. In contrast to the Langmuir isotherm, which was based on the idea that the amount of adsorption does not change, the empirical Freundlich equation, which is based on the idea that the amount of adsorbed material decreases logarithmically with how many sites are filled [43-45].

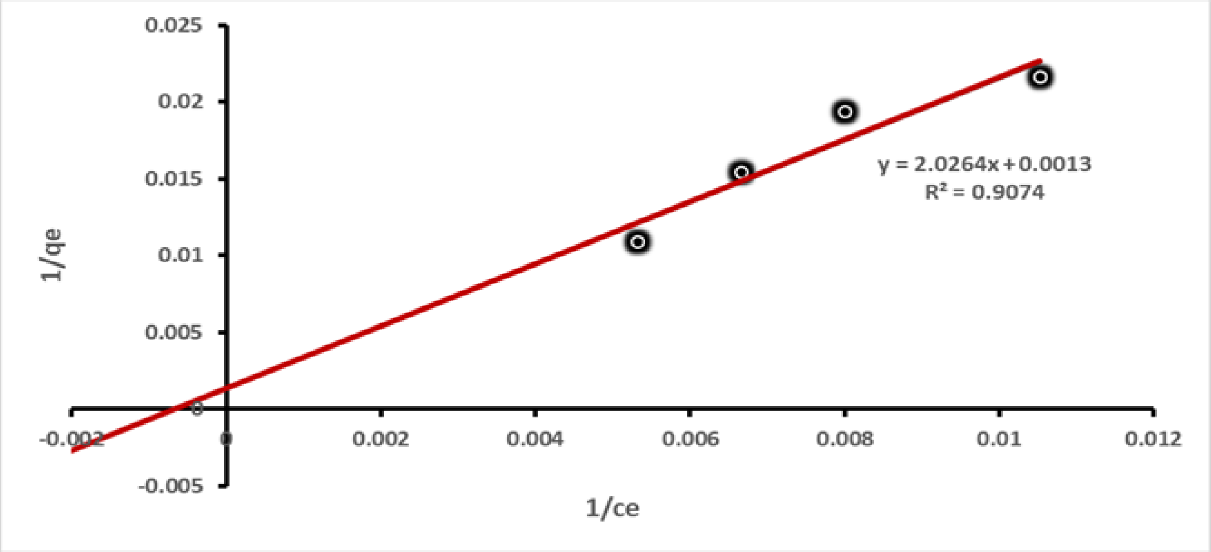

**Fig. 4.** Correlation diagram of adsorption isotherm of Langmuir

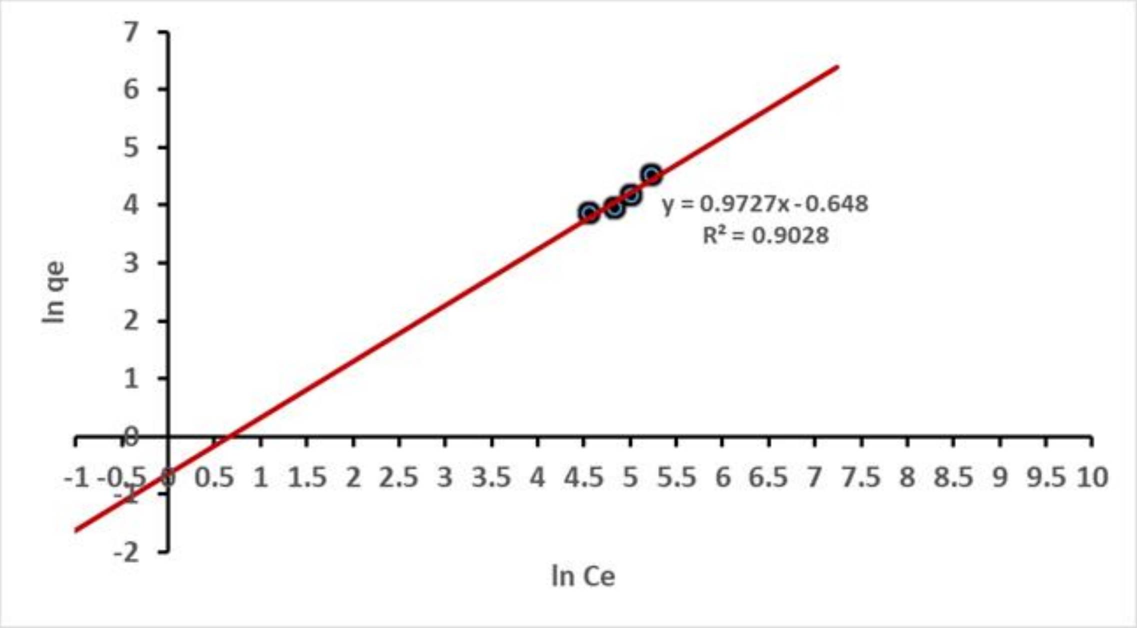

**Fig. 5.** Correlation diagram of adsorption isotherm of Freundlich

**Table 2**
The calculated value of Langmuir and Freundlich for sodium-ion adsorption onto zeolite A

| Langmuir | | | | Freundlich | | |
|---|---|---|---|---|---|---|
| $R^2$ | $K_L$ | $q_m$ | $R_L$ | $R^2$ | $K_f$ | 1/n |
| 0.9074 | 6.4153 x $10^{-4}$ L/mg | 769.3 mg/g | 0.847 | 0.9028 | 0.5231 (mg/g) (1/mg) 1/n | 0.97087 |

The primary properties of the Langmuir isotherm can be expressed as unilateral separation factor constants or equilibrium parameters ($R_L$) by Eq. (3) in Section 2.1, where the $R_L$ value of the Langmuir isotherm is non-reversible when ($R_L = 0$), favoured when ($0 < R_L<1$). The $R_L$ value for sodium-ion concentration adsorption is 0.847, which defines the adsorption as being preferred. This implies that good adsorption occurs throughout the range of pollutant concentrations [42,46,47]. For the Freundlich isotherm, the favourable adsorption of this model may be described in such a manner that if a value for n is greater than unity, adsorption is favourable, and the model undergoes a physical process [48-51]. According to the results of this investigation, the value of n (n=1.03) is more than 1, which indicates that the adsorption mechanism is favourable. Though it is a bit lower than the value of the Langmuir isotherm, the correlation coefficient ($R^2 = 0.9028$) is still rather high.

### *4.5 Adsorption Kinetic*

Comparing the $R^2$ enclosed in value, one will demonstrate the applicability of sodium ion adsorption to zeolite-A in seawater solution. The pseudo-first order in Figure 6 is a plot by ln ($q_e$- $q_t$) against time. The pseudo-first-order analysis gives the $R^2$ value to be 0.732. It reveals that the value of $R^2$ is low. The $R^2$= 0.732 values indicated that the 73.2 per cent efficiency of sodium-ion adsorption capacity towards zeolite-A at a time, $q_t$, depends on the change in the time (min) value. From the straight-line equation, Y= mX + C, the gradient value represents the PFO constant value, $K_1$, whereas the value of C represents the value of $q_t$. A plot $1/q_t$ versus 1/t for the pseudo-second order is outlined in Figure 7. From the plotting, the $R^2$ value is 0.832, higher than the pseudo-first-order value. The calculated pseudo-second-order value of $q_e$ is 104.1 mg/g, near the experimental value of $q_e$ (92 mg/g). The value of $R^2$= 0.832 indicates that the efficiency of 82.2 per cent of sodium ion adsorption against zeolite-A, $q_t$, depends on the time (min) value varies. Pseudo-second-order adsorption suggests an action rate that can be controlled by chemical adsorption involving valence by sharing or exchanging electrons between the materials absorbed by the adsorbent, in addition to physical adsorption [46]. The findings obtained are consistent with the discoveries of some other researchers where the second pseudo-order model is considered to be the most suitable kinetic model compared to the pseudo-first order for the adsorption processes [52,53].

Figure 8 shows a plot of $q_t$ against $t_{0.5}$ for the Intra particle diffusion model. The $R^2$ number is closer to one when compared to the pseudo-first and pseudo-second orders. The adsorption of sodium ions from seawater was found to be appropriate and to best fit this [54-57]. The calculated value of $q_e$, for intraparticle diffusion, which is ($q_e$ = 88.4mg/g was the nearest to the experimental value of $q_e$ (92 mg/g). Furthermore, the value of $R^2$= 0.9117 indicates that 91.17 per cent of sodium ion adsorption efficiency against zeolite, $q_t$ depends on the various time (min) values. The comparison of constant values for pseudo-first-order, pseudo-second order, and intra-particle diffusion as in Table 3 was listed.

By comparing the coefficient of determination, $R^2$ and the adsorption capacity of sodium-ion at equilibrium, $q_e$ for pseudo-first-order, pseudo-second order and intra-particle diffusion, it can be inferred that the intra-particle diffusion model was better adapted for the adsorption of sodium ion to zeolite-A with a higher $R^2$ value (0.9117) and $q_e$ for the conducted experiment is (88.4 mg/g). The intra-particle diffusion process reveals that the sodium ion would have to be diffused through the bulk of the solution to the zeolite-A layer and then through the zeolite-A mesoporous film [54].

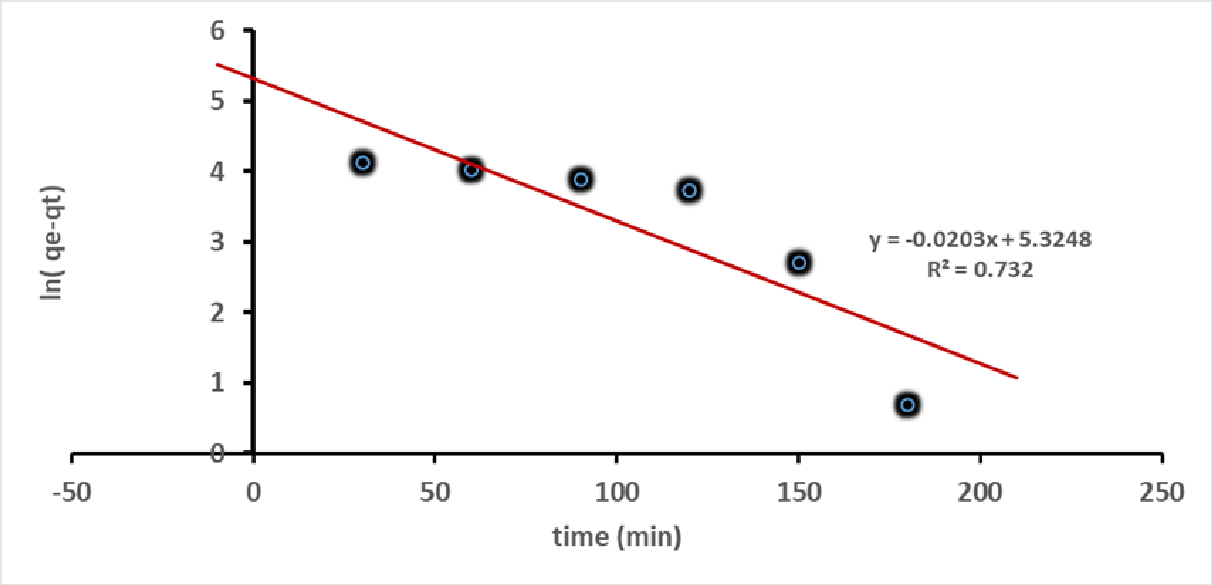

**Fig. 6.** The correlation diagram of adsorption kinetic (Pseudo-First-Order)

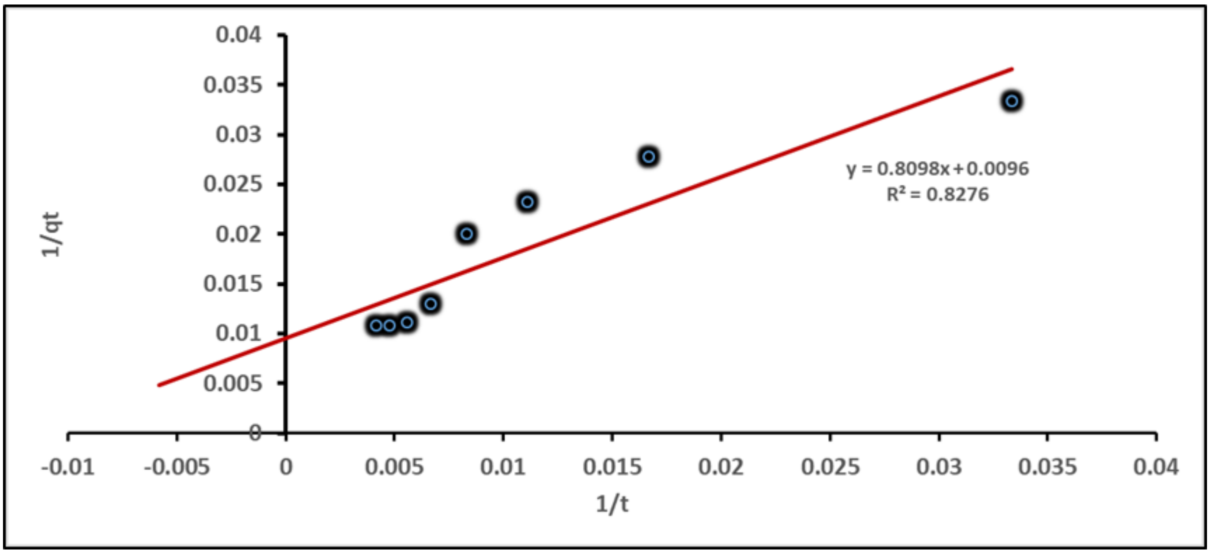

**Fig. 7.** The correlation diagram of adsorption kinetic (Pseudo-Second-Order)

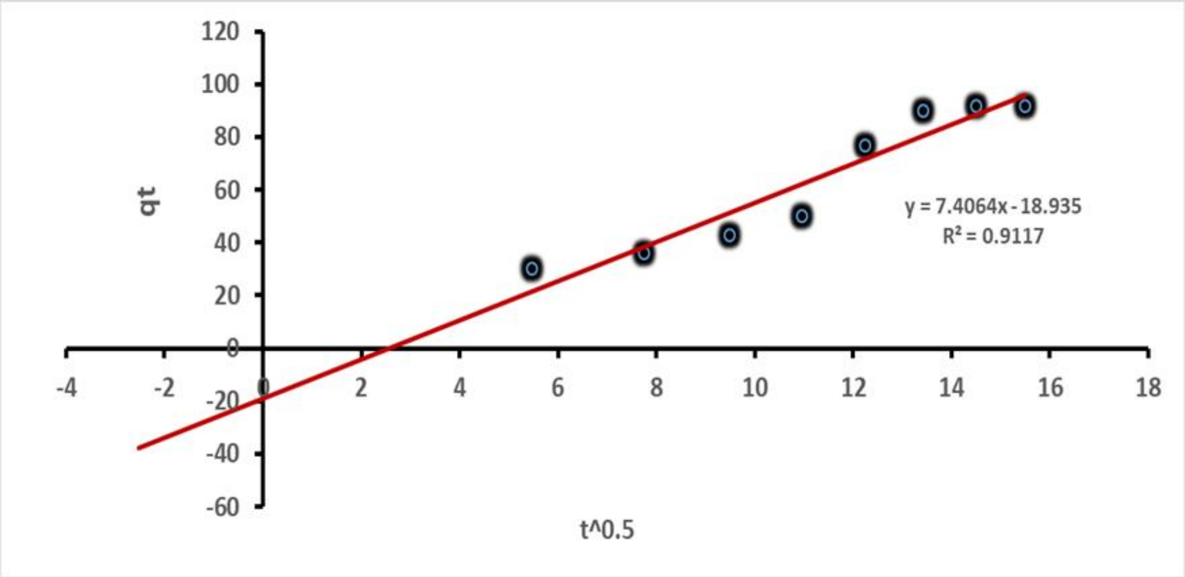

**Fig. 8.** The correlation diagram of adsorption kinetic (Intra Particle)

**Table 3**
The calculated value of kinetic model

| Kinetic model | Constant | Linear regression seawater |
|---|---|---|
| PFO | $R^2$ | 0.732 |
| | $K_1$ ($min^{-1}$) | 0.0203 |
| | $q_e$, (cal) (mg/g) | 205.36 |
| PSO | $R^2$ | 0.8276 |
| | $K_2$ (g/mg min) | 0.0001138 |
| | $q_e$, (cal) (mg/g) | 104.1 |
| ID | $R^2$ | 0.9117 |
| | $K_{id}$ (mg/g $min^{0.5}$) | 7.4064 |
| | C | 18.935 |
| | $q_e$, (cal) (mg/g) | 88.4 |
| Experimental | $q_e$, (exp) (mg/g) | 92 |

## 5. Conclusions

This study discovered that the kaolin-derived zeolite A demonstrated excellent adsorption of sodium ions from seawater solution. Adsorption was observed to be effective with the synthesised Zeolite-A. The results of the batch experiment confirmed that zeolite-A could be used as an adsorbent to remove sodium ions from seawater solution. At equilibrium, zeolite A had a maximum sodium-ion adsorption capacity of 92 mg/g. To depict the adsorption characteristics of zeolite-A, the Langmuir and Freundlich models were used. Compared to the Freundlich model, the Langmuir model produced a considerably excellent performance. The correlation coefficient, $R^2$, for the Langmuir isotherm was 0.9074 and for the Freundlich isotherm was 0.9028. The kinetic analysis indicated that zeolite-A was favourable for the adsorption of sodium ions from seawater solution. When compared to Pseudo first order ($R^2$ = 0.732) and Pseudo second order ($R^2$ = 0.8276), the $R^2$ value of the Intra particle diffusion model ($R^2$= 0.9117) suggested a better-suited outcome. When compared to Pseudo First Order ($q_e$ = 205.36 mg/g) and Pseudo Second Order ($q_e$ = 104.1 mg/g), the value of the adsorption capacity at equilibrium, $q_e$, using Intra particle diffusion model ($q_e$ = 88.4 mg/g) shows the closest qe value from experimental data ($q_e$ = 92 mg/g). In the study, it was found that the adsorption of sodium ions onto Zeolite-A is a very complex process. The efficiency of the adsorption process is determined by the boundary layer diffusion caused by kinetic control of the chemical reaction itself, external mass transfer and intraparticle diffusion inside the exchanger.

**Acknowledgement**

The researcher would like to acknowledge the Universiti Tun Hussein Onn Malaysia (UTHM) for the GPPS financial support grant no H696 and the Internal Grant RDU210314 (Universiti Malaysia Pahang).

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