



Decontamination and adsorption modelling of aqueous Pb^{2+} and Co^{2+} ions using natural inorganic materials: tripoli (NT) and bentonite (NB)

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ABSTRACT

Decontamination and adsorption modelling of Co^{2+} and Pb^{2+} ions from a model aqueous solution have been studied in batch system using natural tripoli (NT) and bentonite (NB). The adsorption study is controlled by a number of factors such as adsorbent dosage, contact time, initial metal ion concentration, and temperature effect. The adsorption equilibrium is achieved during the first 60 min. The maximum removal 97% (approx.) is obtained at 10 g/L and 20°C. The dynamic isotherm has been successfully modelled by the Langmuir ($R^2 = 0.98$). The negative thermodynamic parameter ΔG indicates for the spontaneous adsorption. The pseudo-second-order kinetic model has much more reasonable for metal ions adsorption process ($R^2 = 1.0$ approx.).

Keywords: Lead; Cobalt; Tripoli; Bentonite; Langmuir isotherm; Adsorption modeling; Pseudo-second order

1. Introduction

Heavy metals are generally considered the most contaminant in the ecosystem. In particular, the presence of lead and cobalt in the environment has led to a number of environmental problems. The high level of these metals must be reduced to achieve the standard concentration before discharging to the environment. The discharge of cobalt and lead in the environment is a matter of concern for both toxicological and esthetical reasons [1–4]. According to the European Union (EU) has stated the limited standard concentration for lead (10 $\mu\text{g/L}$) and cobalt (below 5 $\mu\text{g/L}$) ions [5,6]. In

general, the heavy metals may be introduced into the aqueous system by many industrial processes that involve metal in their productive cycle [7].

In order to solve the problem of heavy metals in the ecosystem, many treatment processes have been applied. The most common methods are precipitation, ion exchange, solvent extraction, photoextraction, ultrafiltration, reverse osmosis, electrodialysis, and adsorption [8].

Generally, the ion exchange and sorption by using natural adsorbents are reported to be the potential alternative for removing the heavy metal ions from aqueous mixtures. Recently, these alternatives have been chosen due to several reasons, for example, it is easy to be handled, low cost, and safe for the

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environment and at the same time has good adsorption properties [9,10]. There are many publications deal with several kinds of natural adsorbents, but at the same time, they do not meet all criteria to be effective adsorbent. Among these adsorbents are mostly not cost effective or/and have limiting resources, for instance using date-pits, spent animal bones and Keratin-composed biosorbents [11–20]. Utilization of NT and NB minerals for removing heavy metals from the aqueous medium are considered one of the most promising candidates in decontamination and disposal of high-level heavy metal wastes [21].

Recently, the adsorption of Co^{2+} on bentonite, alumina and silica [22,23] and zeolites [24] have been reported. These sorptions have been applied by a number of factors including pH. These studies have been found that sorption of Co^{2+} were strongly dependent on pH. In addition, the removal of Pb^{2+} from aqueous solution using natural phosphate [25], Na-rectorite [26], and MX-80 bentonite [27] have been also controlled by pH and dosage concentration.

The previous researches have reported the adsorption of Co^{2+} and Pb^{2+} on bentonite and silica in pH effect, wherein, the sorption of metal ion on mineral is enhanced at low pH values [22]. The objective of this work is to study sorption characteristics of Pb^{2+} and Co^{2+} onto the NT and NB. The adsorbing metal ions in solution are demonstrated as a function of dynamic condition parameters, which are different from the previous researches. The adsorption is controlled by a number of factors such as adsorbent dosage, contact time, initial metal ion concentration, and temperature effect. Values of thermodynamic and isotherm models studies for adsorption of these heavy metal ions onto NB and NT have been also investigated and reported. The present work also aims to examine the ability of two existing rate models (pseudo-first-order and pseudo-second-order) for quantifying and predicting the sorption behavior of these heavy metals onto NB and NT.

2. Materials and methods

2.1. Natural inorganic sorbent material

The natural tripoli (NT) and natural bentonite (NB) were supplied from the Directorate of Laboratories in the Natural Resources Authority of Jordan. It was grinded and then screened into particle size of 75 μm using standard Tyler screen series. In order to remove carbonate and other impurities, chemical treatment was performed by the addition of 0.1 M HCl, 0.1 M HNO_3 , and 0.1 M NH_4Cl to the samples of NB and NT fractions. The samples were finally washed with

distilled-deionized water. After 24 h, the solid phases were separated from the solution using centrifuge and then oven-dried at 120°C. Afterwards; the materials were kept under vacuum for several days at room temperature. The structural and the physicochemical properties of Jordanian NT and NB were published elsewhere [28,29].

2.2. Reagents

All chemicals used were analytical grade and were used without further purification. The metal salts of ($\text{Co}(\text{NO}_3)_2 \cdot 3\text{H}_2\text{O}$ and $\text{Pb}(\text{NO}_3)_2 \cdot 3\text{H}_2\text{O}$) were commercially purchased from Fluka Chemika. NaOH, HNO_3 and HCl were purchased from Merck.

2.3. Apparatus and instrumentation

A version 55B atomic absorption spectrometer was used to analyze dissolved Co^{2+} and Pb^{2+} ions. A calibration curve was established for each metal ion. The mixtures were prepared by a thermostatic mechanical shaker at constant temperature (20, 30, and 40°C) of a model (Isothermal Gefellschaft Fur 978). To ensure accuracy in preparation, analytical balance is used (model of Sartorius, CP324-S).

2.4. Sorption studies and measurements

2.4.1. Effect of contact time:

The adsorption experiments were carried out by shaking 0.2 g of the NB or NT with 50 mL of 100 ppm of the heavy metal solution. They were shaken by a thermostatic shaker at constant temperature (20, 30, and 40°C). The agitation speed was fixed at 300 rpm for time ranging between 0 and 150 min. The stirring was kept constant for each run throughout the experiment to ensure equal mixing. At the end of the predetermined time interval, the adsorbent was removed by filtration, and then a sample of the filtrate is analyzed by means of the Atomic Absorption Spectrometer (AAS). It should be noticed that the pH of the solution was adjusted as original stock solution pH.

2.4.2. Effect of dosage

Adsorption measurements were made by a batch technique at temperature of $20 \pm 1^\circ\text{C}$, where a thermostatic mechanical shaker fixed the temperature. Different masses ranged from 0.1 to 1.0 g of NT and NB were placed in a 100 mL stopper plastic flask containing 50 mL of 100 ppm metal ion solution without any change in the pH of the stock solution. The solutions were shaken vigorously using thermostatic mechanical shaker for a given time period (150 min) to reach the

equilibrium. The agitation speed (300 rpm) was kept constant for each run to ensure equal mixing. After completion of a pre-selected shaking time, the suspensions were filtered through a Whatman filter paper. Afterwards, the supernatant solution in each flask was analyzed by AAS for residual heavy metal ions content.

2.4.3. Effect of initial concentration

The adsorption experiments were carried out as described above (Section 2.4.1) by shaking 0.2 g of the NB or NT with 50 mL of the aqueous Co^{2+} or Pb^{2+} ions solution in variant initial concentration (20–100 mg/L) at constant contact time (2.5 h), temperature (20°C) and agitation speed (300 rpm).

2.4.4. Effect of temperature

The adsorption experiments were carried out as described above (Section 2.4.1) by shaking 0.2 g of the NB or NT with 50 mL of the 100 ppm Co^{2+} or Pb^{2+} ions aqueous solution at constant contact time (2.5 h) and agitation speed (300 rpm) with variant temperatures 20, 30 and 40°C.

2.5. Data analysis

The uptake of Co^{2+} and Pb^{2+} ions was calculated from the mass balance, which was stated as the amount of metal ions adsorbed onto the NT or NB. It equals the amount of Co^{2+} and Pb^{2+} ions that is removed from the solution. Mathematically, it can be expressed by Eq. (1) [30]:

$$q_e = \frac{(C_i - C_e)}{S}, \quad (1)$$

where q_e is the heavy metal ions concentration adsorbed by an NT or NB at equilibrium (mg of metal ion/g of adsorbent), C_i is the initial concentration of metal ions in the solution (mg/L), C_e is the equilibrium concentration or final concentration of metal ions in the solution (mg/L), S is the dosage (slurry) concentration and it is expressed by the following equation:

$$S = \frac{m}{v}, \quad (2)$$

where v is the initial volume of metal ions solution used (L) and m is the mass of adsorbent.

The adsorption percentage (%) is calculated using the following equation

$$\% \text{ adsorption} = \frac{C_i - C_e}{C_i} \times 100\%. \quad (3)$$

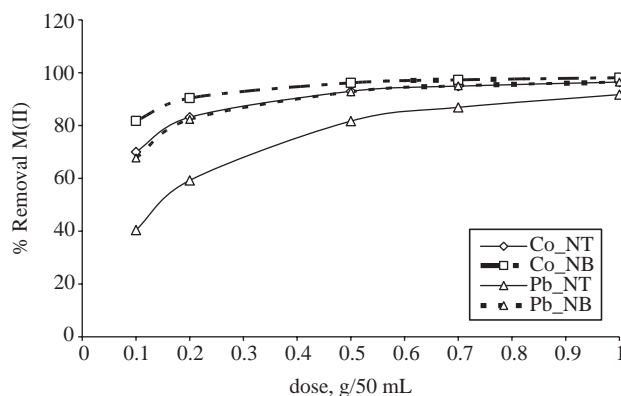


Fig. 1. The effect of variant NB or NT adsorbent dosage (2–20 g L⁻¹) on the removal of Pb^{2+} and Co^{2+} ions from aqueous solution: $t = 150$ min., $\text{pH} = 7$, $T = 20^\circ\text{C}$, $\text{rpm} = 300$, and $C_i = 100$ ppm.

2.6. Kinetic modeling

The pseudo-first-order and pseudo-second-order kinetic equations are used as described in the literatures [31,32].

3. Result and discussion

3.1. Dosage effects

Fig. 1 shows the dosage effect of NT or NB on the removal of Pb^{2+} and Co^{2+} . The adsorbent dosages are varied from 2.0 into 20.0 g/L. The removal percentage of heavy metals increases with increase in the adsorbent concentration. This could be attributed to the increasing of the adsorbent surface area and availability of more adsorption sites. The removal percentages for Pb^{2+} ions on NB or the Co^{2+} ions on NT have the same performance in whole adsorbent doses. The maximum percentages of removal have been calculated in value of 95% (approx.) at 1 g/50 mL (20 g/L) of adsorbent. The removal percentage of Pb^{2+} or Co^{2+} using NB is slightly found higher than NT.

3.2. Temperature effect

The temperature effect considers one of the factors that indicate the type of the adsorption mechanism. The adsorption mechanism is often an important indicator to describe the type and the level of interactions between the adsorbate and adsorbent. If the adsorption decreases with increasing the temperature, this may be an indication for the physisorption; and in general the reverse is true for chemisorptions [25].

The effect of temperature on the removal of Pb^{2+} and Co^{2+} from aqueous solution by using NT and NB has been studied through the varying in the temperatures between the ranges of 20–40°C. Fig. 2 shows

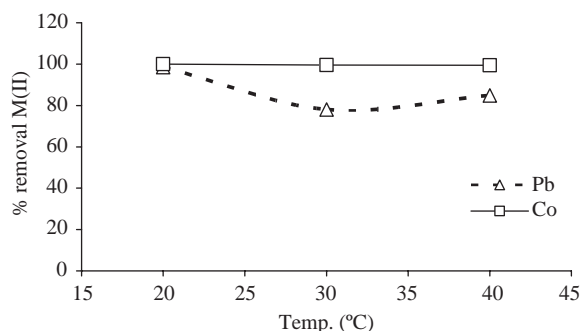


Fig. 2. The temperature effect on the removal of Pb^{2+} and Co^{2+} ions from the aqueous solution by NB: pH = 7, $T = 20\text{--}40^\circ\text{C}$, $C_i = 100$ ppm, $t = 150$ min, and rpm = 300.

the adsorption of Pb^{2+} or Co^{2+} ions on NB in three values of temperatures. The removal percentage decreases with increase in temperature, which could be an indication for physisorption mechanism. The maximum percentages of removal that could be achieved at 20°C for both metals have been calculated around 99%. The adsorption of Co^{2+} ions on NB surface has been found non-highly affected by raising the value of temperatures. This indicates to the weakly physisorption interaction of Co^{2+} ions with NB surface. This type of interaction is considered the typical bio-sorption for the most of the aqueous metal ions [33].

Fig. 3 shows the maximum percentages of the aqueous Pb^{2+} ions removal on NT surface, which initially increases with increase of the temperature values. But then, the process slow down and subsequently attain a constant value in a high temperature value at 40°C . Also, the figure shows the maximum percentages of the aqueous Co^{2+} ions on the NT surface, which decreases with increase the value of temperatures. This is because

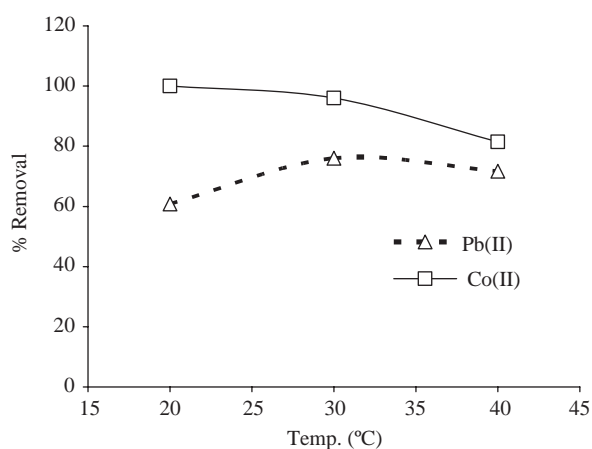


Fig. 3. The temperature effect on the removal of Pb^{2+} and Co^{2+} ions from the aqueous solution by NT: pH = 7, $T = 20\text{--}40^\circ\text{C}$, $C_i = 100$ ppm, $t = 150$ min, and rpm = 300.

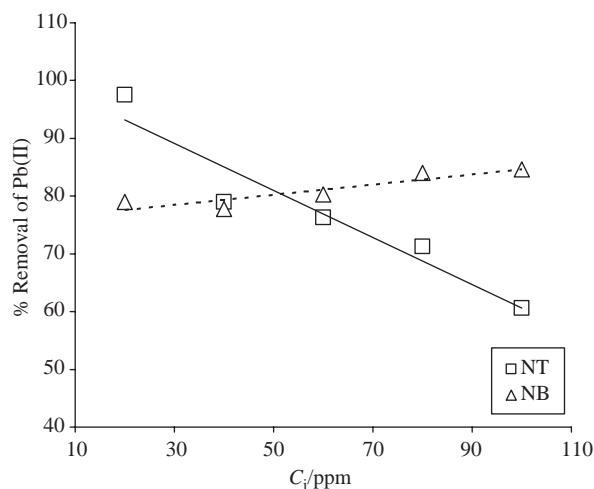


Fig. 4. The initial concentration effect of Pb^{2+} ion on the removal efficiency using NT and NB: 4g (NT or NB)/L, $T = 20^\circ\text{C}$, $t = 150$ min, and rpm = 300.

with increasing temperature, the attractive forces between NT surface and Co^{2+} ions are weakened and the sorption start to decrease. At high temperature, the thickness of the boundary layer decreases. This is due to the increased tendency of the metal ion to escape from the NT surface to the solution phase, which results in a decrease in adsorption as temperature increases [34].

3.3. Effect of metal initial concentrations

To evaluate the effect of initial concentration on the removal efficiency, the solution of Pb^{2+} or Co^{2+} ions are varied in the range of 20–100 mg/L, and then all other factors are kept constant.

Fig. 4 shows the removal percentage for the aqueous Pb^{2+} ions by the adsorption on the surfaces of both of the NT and NB, which results from the change in initial concentration of metal ion. The removal percentage for the aqueous Pb^{2+} ions by the adsorption on NT sharply decreases with increase in initial concentration of metal ions, while the reverse is noted using NB. This is due to the strong interactive forces between Pb^{2+} ions and NT, and the reverse is suggested in case of using NB. This phenomenon has been confirmed by many reported researches [35], where the adsorbed amount of Pb^{2+} ions decrease in case of using kaolinite and montmorillonite. This is due to the presence of high adsorbent amount that creates the aggregation of particle. These aggregations could cause a decrease in the total surface area and an increase in diffusion path length of which contribute in decreasing of the adsorbed amount per unit mass [35].

Fig. 5 shows the removal percentage for the aqueous Co^{2+} ions by the adsorption on the surfaces of

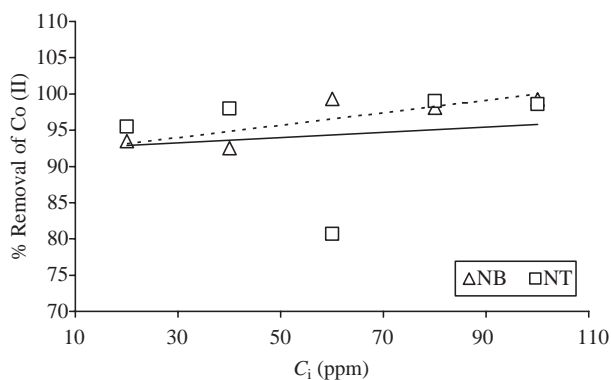


Fig. 5. The initial concentration effect of Co^{2+} ion on the removal efficiency using NT and NB: 4g (NT or NB)/L, $T = 20^\circ\text{C}$, $t = 150$ min, and $\text{rpm} = 300$.

both of the NT and NB, which results from the change in initial concentration of metal ion. The removal percentage for the aqueous Co^{2+} ions on the surfaces of the NT or NB increases with increase of the initial metal concentrations. The maximum percentage of aqueous Co^{2+} ions removal has been obtained around 96% by both adsorbents. This could be an indication for a weak interaction between Co^{2+} and adsorbent (NB or NT).

3.4. Isotherm modeling

In this study, the experimental isotherm data set obtained was fitted using the Langmuir adsorption model. The Langmuir adsorption [36] is based on the theoretical principle that only a single adsorption layer exists on an adsorbent and represents the equilibrium distribution of metal ions between the solid and liquid phases. The basic assumption of the Langmuir adsorption process is the formation of monolayer of adsorbate on the outer surface of the adsorbent and after that no further adsorption takes place.

The Langmuir-type isotherm remains to be the most widely used for practical applications. The Langmuir isotherm for pure component adsorption is demonstrated in the following equation:

$$\frac{C_e}{q_e} = \frac{1}{q_{\max}b} + \frac{1}{q_{\max}}C_e, \quad (4)$$

where q_{\max} is the maximum metal ions uptake per unit mass of adsorbent (mg/g) which is related to adsorption capacity and b is Langmuir constant (L/mg) which is exponentially proportional to the heat of adsorption. Therefore, a plot of C_e/q_e vs., gives a straight line of slope $1/q_{\max}$ and intercept $1/(q_{\max}b)$ as shown in Fig. 6.

The calculated results of the q_{\max} are listed in Table 1. The high values of correlation coefficients, R^2 , is greater than 98 for both adsorbents which might

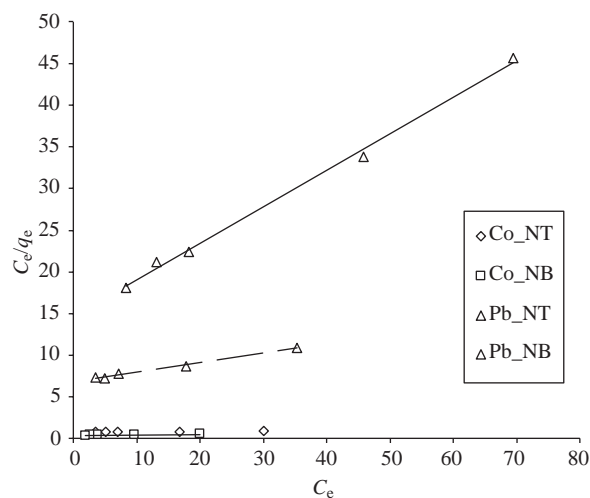


Fig. 6. The linearized Langmuir adsorption isotherms for the adsorption of Pb^{2+} and Co^{2+} ions over NT and NB: $C_i = 100$ ppm, $T = 20^\circ\text{C}$, $t = 150$ min, $T = 20^\circ\text{C}$, and $\text{rpm} = 300$.

be attributed to that the adsorption of Pb^{2+} and Co^{2+} onto both NT and NB can be well described using the Langmuir isotherm.

It can be observed that the q_{\max} values, which are generated from NB for both metal ions, are greater than those values that are generated from NT. This means that the NB is more powerful adsorbent than NT. Among the studied heavy metals, we observed that q_{\max} of $\text{Co}^{2+} > \text{Pb}^{2+}$ for both of NB and NT. In order to justify the validity of NT and NB as an adsorbent for Pb^{2+} and Co^{2+} ions adsorption, its adsorption potential must be compared with other adsorbents like zeolite and activated carbon. It may be observed that the maximum sorption of Pb^{2+} on NT is approximately the same to those on the activated carbon. While, the maximum sorption of the same metal on the NB is approximately the same to those reported of zeolites [24]. In contrast to the reported adsorbent of natural zeolite [37], we found that the adsorption of Co^{2+} using both NT and NB is much higher 3 and 4 times, respectively.

The very useful relationship between standard free energy change (ΔG°) and the Langmuir constant, b is given by the following equation [38,39]

$$\Delta G^\circ = -RT \ln K, \quad (5)$$

wherein R is universal gas constant (8.314 J/mol K) and T is the absolute temperature in Kelvin and K is the equilibrium constant, related to the Langmuir constant, b via Eq. (6)

$$K = b \cdot M_A, \quad (6)$$

where M_A is the molar weight of sorbate

Table 1
Langmuir constants and thermodynamics parameters at 20°C

Langmuir constants Adsorbent Type Metal ion	q_{\max} (mg/g)		$K = bM_A$ (L/mol)		ΔG (kJ/mol)	
	NT ¹	NB ²	NT	NB	NT	NB
Co ²⁺	217.4 ($R^2 = 0.99$)	270.3 ($R^2 = 0.98$)	317.5	570.6	-14.7	-15.7
Pb ²⁺	50 ($R^2 = 0.98$)	125 ($R^2 = 0.99$)	2,325	2,332	-19.2	-19.2

1. NT = Natural Tripoli. 2. NB = Natural Bentonite.

The values of K and ΔG^0 for the adsorption process were calculated and they are also listed in Table 1. The negative value of ΔG^0 confirms the feasibility of the process and the spontaneous nature of sorption with a high preference for metal ions to be adsorbed onto NT and NB.

3.5. Kinetic modeling

Quantifying the changes in the sorption with time requires that an appropriate kinetics models to be used. The adsorption kinetic of metal ions using NT and NB was analyzed using various kinetics models.

The effect of adsorption time on the removal of both metal ions using NT and NB was studied using metal ion solutions (100 mg/L) with $V/m = 50 \text{ mL}/0.2 \text{ g}$ of NT or NB. The prepared solution is used without change in its normal pH. Samples were collected at regular intervals and then analyzed after filtration.

The kinetic profiles of these heavy metals are shown in Fig. 7 for both of the adsorbents. It is clearly shows that the adsorption rate initially increased rapidly and then the equilibrium concentration is attained within 60 min. After this equilibrium period, the amount of adsorbed of both metal ions did not significantly changed with

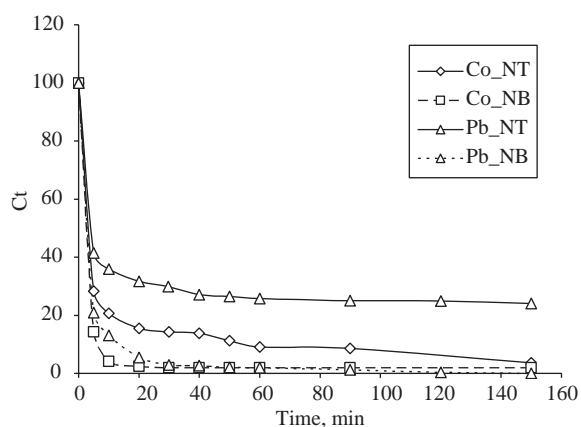


Fig. 7. The contact time effect on the removal of Pb²⁺ and Co²⁺ ions in the interval of 5–150 min: $C_i = 100 \text{ ppm}$, dosage = 4 g/L, $T = 20^\circ\text{C}$, and rpm = 300.

time. Fig. 7 shows a rapid transfer of metal ion from solution to the solid adsorbent within 5 min. The final percentages (% C_t) using NB are calculated and found 21% and 14% for Pb²⁺ and Co²⁺, respectively. While they are calculated using NT and found 41% and 28% for Pb²⁺ and Co²⁺, respectively. The results indicate that the NB has stronger ability to adsorb Pb²⁺ and Co²⁺ more than using NT. These results are closed to what obtained from Langmuir constant q_{\max} (Table 1). The adsorption rate of Pb²⁺ ion using NB is found greater than using Na-rectorite [26]. A similar adsorption behavior has been recently confirmed by Donate et al. [40] and Xu et al. [27].

The data were then regressed against the pseudo-first-order and pseudo-second-order kinetics models. The slopes and intercepts of these curves were used to determine the pseudo-first-order and pseudo-second-order constants k_1 and k_2 and the equilibrium capacity q_e as shown in Table 2.

In general, the adsorption rate constant shows that adsorbate is adsorbed faster by the adsorbent. According to adsorption rate constants as shown in Table 2, both metal ions are faster adsorbed on the NB than NT for Lagergen pseudo-first-order kinetics model. This was confirmed by equilibrium contact times determined from their kinetic curves. For the case of the second-order kinetic model, both metal ions are faster on the natural NB than NT.

Table 2
The adsorption kinetic model rate constants for adsorption of Co²⁺ and Pb²⁺ ions on NT and NB

	Pseudo-first-order		Pseudo-second order		
	k_1	R^2	k_2	q_e	R^2
Co ²⁺					
NT	0.026	0.7	0.057	4.86	0.998
NB	0.131	0.8	0.781	4.92	1
Pb ²⁺					
NT	0.032	0.8	0.118	3.80	1
NB	0.037	0.8	0.138	5.04	1

4. Conclusion

Natural inorganic materials such as NT and NB can be used for the removal of aqueous Pb^{2+} and Co^{2+} . The removal percentage of metal ions is increased as the dosages increased. The maximum removal of ions on both NB and NT could be achieved at low temperature as physisorption mechanistic, while the behavior of Pb^{2+} on NT has undefined behavior. The removal percentage increases as the initial concentration increases, however the opposite behavior was observed for Pb^{2+} on NB. The results from this study were extremely well described by the theoretical Langmuir isotherm, wherein R^2 is greater than 98. The theoretical Langmuir of the maximum metal ion uptake (q_{max}), which are generated from NB for both metal ions, are greater than those generated values from NT for the same metal ions. In addition, the q_{max} values of $Co^{2+} > Pb^{2+}$ for both NT and NB. We can conclude from the adsorption kinetic model that both metal ions are faster adsorbed on the NB than NT using Legeergen pseudo-first-order and second-order models.

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