

Comparative Study to Assess Phosphorus Removal from Wastewater Using Aluminum Modified Clay and Chitosan Lanthanum Adsorbents.

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Abstract: This study compares the efficiency of removal of phosphorus from wastewater using two different adsorbents. The first adsorbent is a clay based adsorbent, and the second one is chitosan based adsorbent. The bentonite based adsorbent has been prepared by adding a solution containing hydroxy polycation of aluminum to bentonite suspended in distilled water. The resulting modified clay was filtered and washed many times with distilled water. After filtration, the aluminum-modified clay adsorbent was dried and ground to a suitable size for adsorption experiment. The chitosan lanthanum adsorbent has been prepared by modifying chitosan solution using lanthanum chloride solution. The La (III)/chitosan ratio was chosen to be 112 mg La/g of chitosan. The resulting lanthanum chitosan mixture has been added to a solution containing 6N NaOH to allow the formation of Ct/La flakes. The Ct/La flakes have been collected using a small mesh and washed many times with distilled water. The dried flakes have been used as an adsorbent for phosphorus removal. Results showed that both adsorbents fitted well to the Freundlich and Langmuir adsorption isotherms with R² values above 0.95. It was found that the adsorption capacities for Al-Bentonite and La-chitosan were 10.71 and 17.9 mg p/ gm adsorbent respectively. Kinetic studies showed that the two adsorbents were well presented by the pseudo-second order kinetics. The pseudo second order rate was found to be 0.00543, and 0.0021 for Al-Bentonite, and La-chitosan respectively. The study concludes that Ct Lanthanum is effective adsorbent and more efficient than aluminum modified bentonite

Keywords: Phosphorus Removal, Adsorption, Chitosan Lanthanum Adsorbent, Aluminum Modified Clay, Wastewater Treatment.

1. Introduction

It is well known that phosphorus (P) is an essential nutrient that is important for healthy ecosystems. On the other hand, the excess concentration of P can be detrimental to water bodies. Excess P can be considered as one of the main causes of eutrophication in the receiving water bodies (Dodds & Smith, 2016). P is not naturally occurring in water bodies where P can find its way to the

environment through different anthropogenic sources such as industrial, agricultural and partially treated domestic wastes. There are many strategies that can be applied to deal with excessive concentrations of P in water bodies. One of the best approaches to limit the flow of phosphorus into water bodies through source reduction by taking essential measures towards the balanced application of phosphorus-based fertilizers use in agriculture (Sharpley & Tunney, 2000), controlling P based detergents, domestic food additives (Comber, Gardner, Georges, Blackwood, & Gilmour, 2013) and use of appropriate waste minimization techniques to minimize the phosphorus concentration in industrial effluents (Mayer *et al.*, 2016). Despite all measures for minimizing P loads into wastewater effluents, having a considerable concentration of P in wastewater is unavoidable and eventually must be treated to protect water bodies from excessive algal growth. There are many treatment techniques for P removals such as chemical precipitation, biological removal, ion exchange and adsorption (De-Bashan & Bashan, 2004; Morse, Brett, Guy, & Lester, 1998). There are many factors governing selecting the appropriate method for phosphorus removal such as cost, the flexibility of the process, discharge limits, need for skilled operators and the possibility of P recovery. Adsorption of P is one of the techniques that started to gain popularity. One of the early P adsorption experiments was the use of alum sludge resulting from drinking water treatment works for removal of P from wastewater (Yang, Tomlinson, Kennedy, & Zhao, 2006). Other studies included adsorption using alumina treated with aluminum sulfate (Hano, Takanashi, Hirata, Urano, & Eto, 1997), metal modified bentonites such as Al, Fe, and La (El-Sergany & Shanableh, 2012; A. Shanableh, Enshasi, & Elsergany, 2016; A. M. Shanableh & Elsergany, 2013; Tian, Jiang, Ning, & Su, 2009). In general phosphorus adsorbents can be classified into three main categories as a natural material, industrial by-products, and man-made products (Vohla, Kõiv, Bavor, Chazarenc, & Mander, 2011). This study aims to investigate the use of lanthanum chitosan preparation as an adsorbent for phosphorus removal.

Chitosan is a deacetylated derivative of chitin. Chitin is a natural polysaccharide available in a wide range of living organisms. Recently, chitin and its deacetylated derivative

chitosan have gained significant popularity in industrial, pharmaceutical and environmental applications because they can be commercially extracted from shellfish processing wastes such as shrimp. Chitosan has been used as natural biopolymer in many environmental applications. Chitosan can be used as a coagulant (Huang & Chen, 1996; Jun, Kim, No, & Meyers, 1994), coagulant aid (Bina, Mehdinejad, Nikaeen, & Attar, 2009), removal of metals, and base for many adsorbents.

Lanthanum is a rare earth element that has high selectivity to phosphorus (Wasay, Haran, & Tokunaga, 1996). Because of its selectivity and affinity to phosphorus Lanthanum has been used in the preparation of many adsorbents for phosphorous removal from water and wastewater. Lanthanum oxide has been used in preparing activated carbon fibers loaded with La for phosphorus removal it was found that this novel adsorbent could remove up to 97.5% of phosphorus in the solution. (Zhang *et al.*, 2011) Similarly, Lanthanum oxide was used to modify natural zeolite for the preparation of La-Zeolite adsorbent this adsorbent showed high removal efficiency and high regeneration potential. Regeneration experiments showed that adsorption capacity of the regenerated adsorbent never fell below 90% of the fresh adsorbent. (Ping, Hans-Jörg, Bing, Xiwu, & ZHANG, 2008) Lanthanum modified bentonite (phoslock[®]) was also used for removal of phosphorous from water. Phoslock was developed in the early 1990s by the Commonwealth Scientific and Industrial Research Organization (CSIRO) of Australia. Since the introduction of phoslock, there have been many studies to investigate its efficiency for removal of phosphorous from water. (Robb, Greenop, Goss, Douglas, & Adeney, 2003; Ross, Haghseresht, & Cloete, 2008) incorporation of Lanthanum into clay structures such as bentonite can result in a stable adsorbents where La is locked inside the clay structure which in turn minimize the possibility of La leaching to water. Accordingly incorporation of lanthanum into clay structure such as the case of phoslock is considered to be environmentally safe. (Groves, 2010)

No previous studies have investigated the use of Lanthanum modified chitosan preparations for removal of phosphorus from water; however, previous studies investigated the use of La chitosan for removal of fluoride from water. This study aims at investigating the efficiency of La-modified chitosan to remove phosphorus from water and compare the lanthanum modified chitosan with other preparations such as Al modified bentonite. (Kamble *et al.*, 2007)

2. Material and methods:

a. Preparation of La-modified chitosan

Lanthanum modified chitosan (CtLa) was prepared according to Bansawal *et al.* (Bansawal, Thakre, Labhshetwar, Meshram, & Rayalu, 2009) In a beaker, 20 g of LaCl₃·7H₂O was dissolved in 100 mL distilled water (1 mL of this solution contains 0.2 g of lanthanum chloride). In another 4 beakers, 2 gm of chitosan in each beaker (medium molecular weight from Aldrich Cat No. 448877) was dissolved in 100 mL acetic acid (CH₃COOH) solution

(1%, v/v). The stock solution of Lanthanum chloride was added to the dissolved chitosan a rate of 1.5 mL / 1 gm of chitosan; this is equivalent to 112 mg of La(III) per gram of chitosan. The resulting La-Chitosan solution was stirred with a magnetic stirrer for 3 hours to make sure that Lanthanum was adsorbed on chitosan. Hence the La-Chitosan solution was added dropwise to a 6N solution of Sodium hydroxide under vigorous stirring conditions to ensure well mixing of the added contents. The resulting mixtures were allowed to stabilize overnight. After stabilization, the resulting mixtures were screened using 65 µm to separate the La-chitosan flakes. The separated flakes were washed with distilled water and dried in the oven at 60°C. After drying; the flakes were ground to a uniform size ranging from 75 to 150 µm.

b. Preparation of Al modified bentonite

Aluminum modified bentonite (AlBt) was prepared by adding 250 mL of the aluminum pillaring solution to 10 grams of Bentonite clay (Poudrszan Industrial and Mineral Group, Dubai, United Arab Emirates) the resulting mixture was completed to one liter with distilled water. The addition of pillaring solution resulted in an overall [Al]/Bentonite ratio of 7.8 mmoles Al per gm of Bentonite. The Al pillaring solution was prepared by adding 300 mL of 1M Na₂CO₃ dropwise to 500 mL 0.5M AlCl₃·6H₂O solution at 60° this corresponds to [OH]:[Al] molar ratio of 2.4. After addition of the pillaring solution to the Bentonite clay, the content was mixed overnight. The modified clay was filtered and washed with deionized water many times to get rid of chlorides. The presence of chlorides was tested qualitatively using 0.1 N silver nitrate solution. The filtered clay was dried at 105° and ground to a uniform size from 75 to 150 µm.

c. Phosphate adsorption experiment

The efficiency of phosphate removal was measured using 50 mL of different initial phosphate concentrations ranging from 10 to 50 mg/L treated with 0.1 g of the adsorbent. The initial pH was adjusted at 5. The adsorption experiment was conducted over a period of 9 hours using a lab-line thermal shaker (Model No.3527-6) at 200 rpm and 25°C. At the end of the experiment, the remaining phosphate concentration was measured according to standard methods for the examination of water and wastewater experiment number 4500-P ascorbic acid method. Similarly, the phosphate adsorption kinetics was performed using a 50 mL of phosphate solution with an initial concentration of 20 mg/L treated with 0.1 g of the adsorbent; the experiment was conducted over a period of 6 hours.

3. Results and discussions:

a. Phosphate Adsorption Isotherms

To compare the adsorption efficiency of the two adsorbents used in this study, the experimental phosphate adsorption results were fitted using the Langmuir and Freundlich, isotherms, expressed in Equations 1 and 2 below:

$$Q_e = \frac{Q_m K_L C_e}{1 + K_L C_e} \quad (1)$$

$$Q_e = K_f C_e^{1/n} \quad (2)$$

As shown in equations 1 and 2 the Q_e is the equilibrium adsorption capacity expressed in mg phosphate-P per g of adsorbent used; C_e presents the remaining concentration of phosphate-P in solution at equilibrium (mg/L); Q_m is the theoretical maximum adsorption capacity expressed in mg P per gram of adsorbent used, and K_L is the Langmuir equation constant. For the Freundlich adsorption isotherm, the K_f and n are Freundlich equation constants.

To graphically present Langmuir and Freundlich isotherms, the linear forms should be used. The linear forms of Langmuir and Freundlich equations can be expressed as shown in Equations 4 and 5:

$$\frac{C_e}{Q_e} = \frac{1}{Q_m K_L} + \frac{1}{Q_m} C_e \quad (4)$$

$$\log Q_e = \log K_f + \frac{1}{n} \log C_e \quad (5)$$

The phosphate adsorption isotherms of the two adsorbents are presented in Figure 1. The experimental adsorption data were fitted to the linear forms of the Langmuir and Freundlich models (Equations 4 and 5 respectively), and the isotherms parameters were estimated, as presented in Table 1.

The correlation coefficients (R^2) obtained values suggest that the data were adequately fitted by the two isotherm

models. In both models, the R^2 values were greater than 0.95, which confirms with earlier reported results for most of the phosphate adsorbents. As shown in Table 1 the Q_m values for the CtLa and AlBt were 17.86 and 10.65 mg/g of adsorbent. Apparently, from the Q_m values, the Chitosan lanthanum adsorbent has much greater adsorption capacity compared to aluminum bentonite adsorbent.

The Langmuir isotherm assumes monolayer adsorption onto a surface containing a number of active sites for adsorption with a uniform adsorption power. Based on this assumption, once the active sites are filled, no further adsorption can take place at the adsorbent surface reaching a state of saturation where the maximum adsorption capacity has already achieved (Desta, 2013). For Freundlich isotherm, the K_f and $1/n$ values reflect the adsorption capacity and adsorption intensity, respectively. The $1/n$ value reflects the heterogeneity factor. For good adsorbents the value of $1/n$ ranges from 0 to 1 (El-Sergany & Shanableh, 2012)., if $1/n > 1$ this means that adsorption is a chemical process. Values of $1/n < 1$ reflects that adsorption is a physical process. The values of K_f for chitosan lanthanum and Aluminum Bentonite adsorbents were 9.27 and 5.1 respectively indicating higher adsorption capacity for the chitosan lanthanum compared to Aluminum modified clay. For both adsorbents, $1/n$ values were less than 1 suggesting that adsorption is physical adsorption.

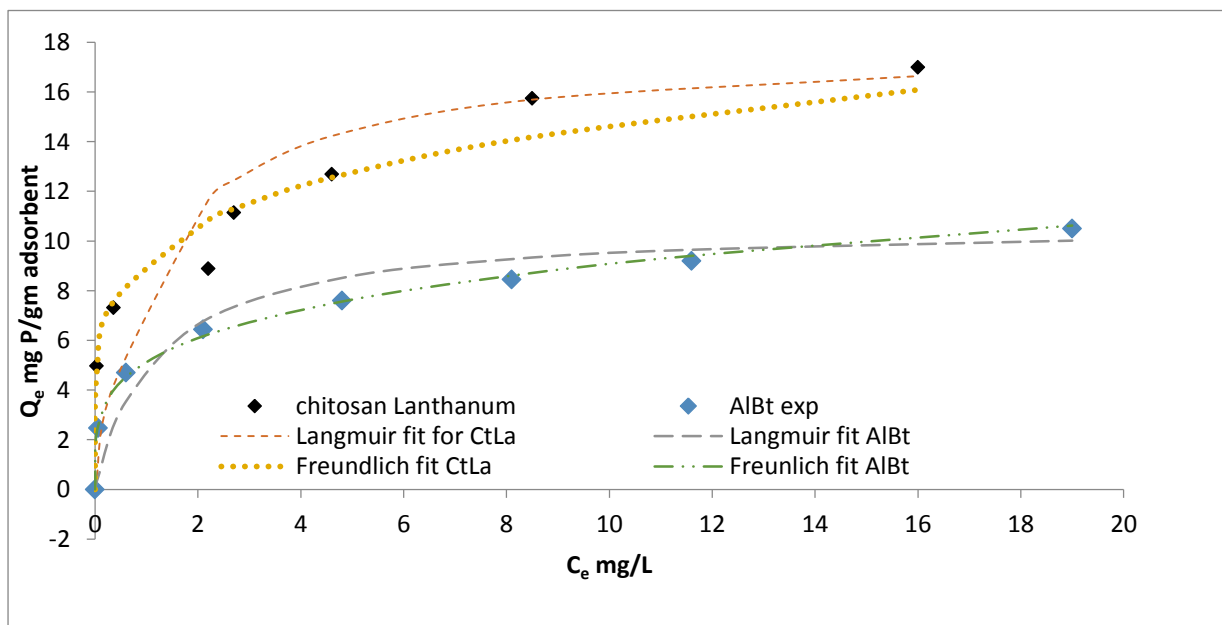


Figure 1. Adsorption isotherm for CtLa and AlBt with experimental data vs the calculated Langmuir and Freundlich model fit

Table 1. Langmuir and Freundlich adsorption constant for CtLa and AlBt adsorbents.

Adsorbent	Langmuir			Freundlich		
	K_L	Q_m	R^2	$1/n$	K_f	R^2
CtLa	0.851	17.86	0.98	0.199	9.27	0.95
AlBt	0.83	10.65	0.99	0.247	5.1	0.99

b. Effect of pH:

The effect of pH on phosphate adsorption removal was studied over a pH range from 5 to 8. This range was chosen because it covers the practical pH range for the majority of water and wastewater samples. The experiment was carried out by using synthetic samples containing initial phosphorus concentration of 10 mg/L, with and an adsorbent dose of 2 g/L. The initial pH was

adjusted to the desired value using 0.1 N NaOH solution. As shown in Figure 2, the phosphate adsorption capacity of both adsorbents decreased as the pH increased. The decrease in the phosphate adsorption capacity is insignificant when pH changed from 5 to 6; however the impact of pH was more significant at pH values greater than 6. 6.

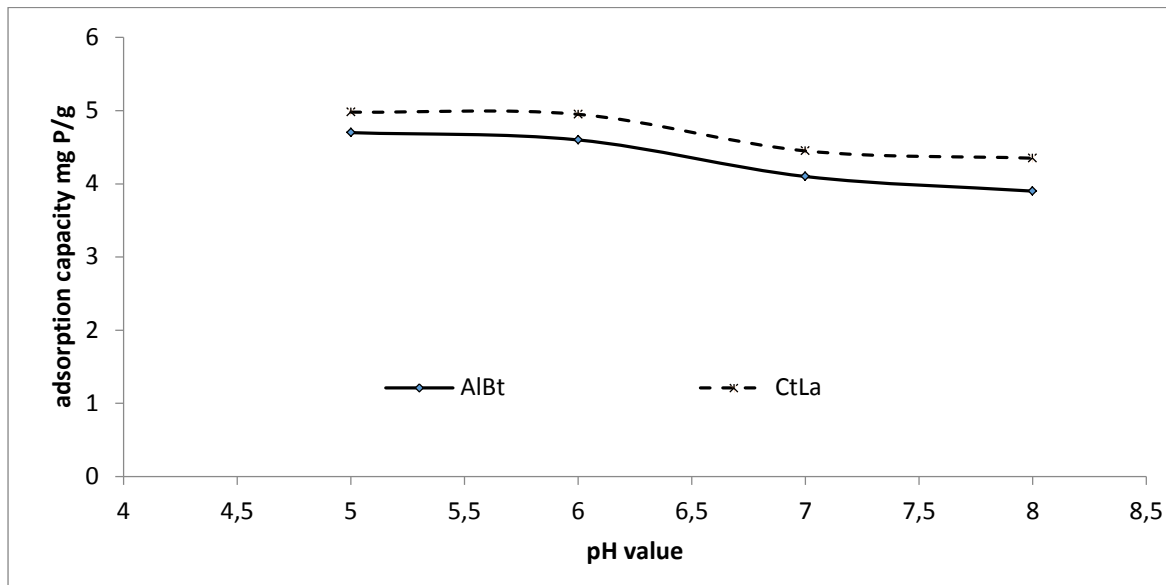


Figure 2. Change of adsorption capacity of both adsorbents at a pH range from 5 to 8

c. Phosphate Adsorption Kinetics

To study the adsorption kinetics, experiments were conducted using synthetic phosphate solutions mixed with 2 g/L of the adsorbent at 25°C and pH 5. The initial phosphate concentration was 20 mg/l and 20.5 mg P/L for CtLa and AlBt respectively. As shown in Figure 3a, the equilibrium adsorption capacity of CtLa is much higher than the AlBt in addition to that the adsorption for AlBt is relatively faster than CtLa. It was found that about 75% and 90% of the equilibrium capacity has been reached in the first two hours of the adsorption experiment for CtLa and AlBt respectively. The status of equilibrium in case of AlBt has been reached within the first 4 hours however the CtLa adsorbent took relatively longer time where equilibrium was reached after 5 hours. In both cases the results are in accordance with previous studies that observed the phosphate adsorption equilibrium is

relatively fast adsorption process where equilibrium is reached from 3 to 8 hours (A. M. Shanableh & Elsergany, 2013; Yan *et al.*, 2010). The kinetic adsorption data were fitted with the linear form of the pseudo-first-order and pseudo-second-order reaction rate models, which are described in Equations 5 and 6 below:

$$\ln(q_e - q_t) = \ln q_e - k_1 t \quad (5)$$

$$\frac{t}{q_t} = \frac{1}{k_2 q_e^2} + \frac{1}{q_e} t \quad (6)$$

where, q_t = phosphorus adsorbed (mg P/g adsorbent) at time t (minutes); q_e = phosphorus adsorbed in mg P/g adsorbent at equilibrium; k_1 = pseudo-first-order rate constant (min^{-1}), and k_2 = pseudo-second-order rate constant in $\text{g}/(\text{mg} \cdot \text{min})$.

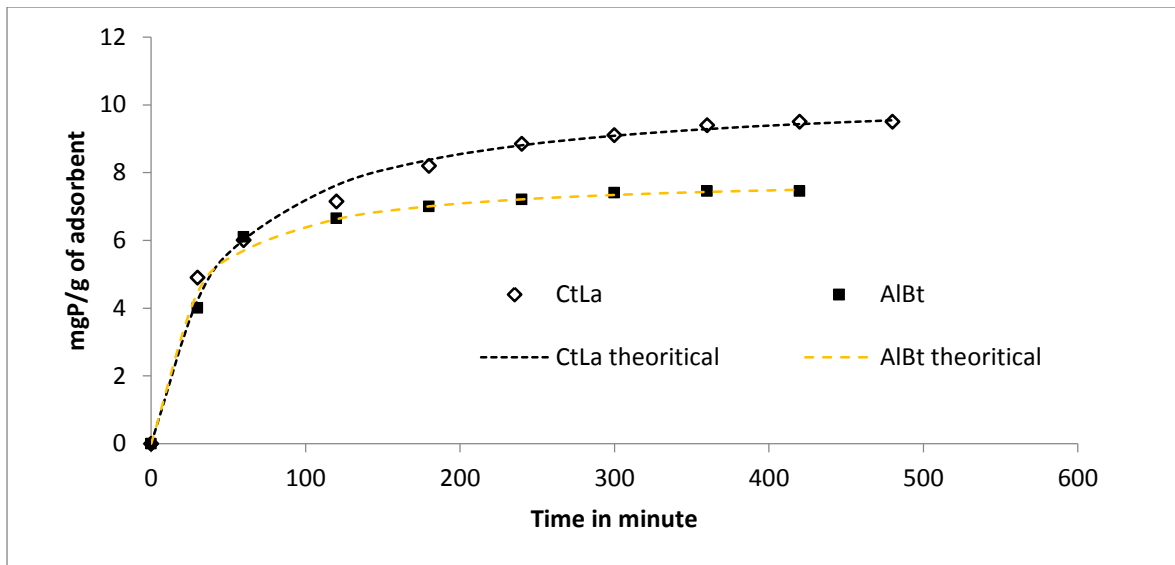


Figure 3. Kinetics of phosphate adsorption onto the two adsorbents fitting with the pseudo-second-order kinetics. (a) adsorption with time; and (b) adsorption data fit with the linear form of the pseudo-first-order reaction, and (c) adsorption data fit with the linear form of the pseudo-first-order reaction

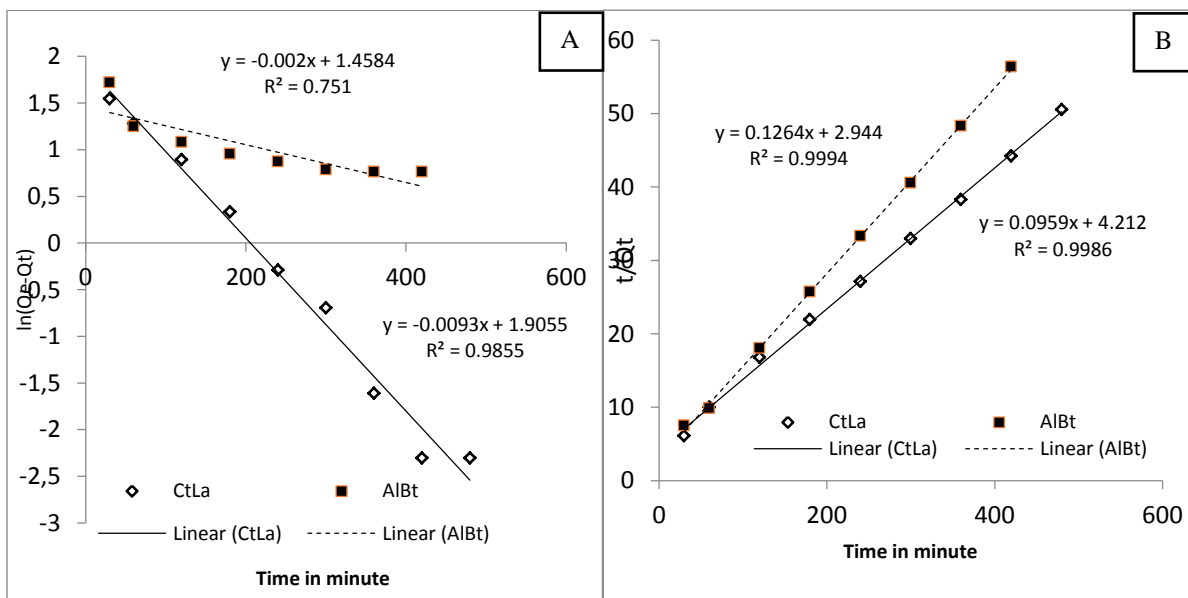


Figure 4. phosphate adsorption kinetics (a) adsorption data fit with the linear form of the pseudo-first-order reaction, and (b) adsorption data fit with the linear form of the pseudo-second-order reaction

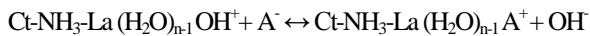
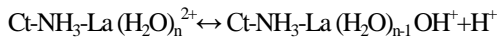
The Kinetic parameters for the pseudo first-order and pseudo second order adsorption models are presented in table 2 and figure 3a and figure 3b. With regards to the R^2 value, It is clear that the experimental data fitted with both the pseudo first-order and pseudo second order for the CtLa adsorbent, however, the data fitted with the pseudo second order adsorption for the AlBt adsorbent. Despite the fitting of the pseudo first order model in the case of the CtLa, the Pseudo first order model failed to predict the value of q_e however the fitting with the pseudo second order model was able to predict the experimental equilibrium adsorption capacity. This finding is in accordance with many other studies that reported that

phosphate adsorption fits better with pseudo second order kinetics more than the pseudo first order kinetics.

d. Adsorption mechanism

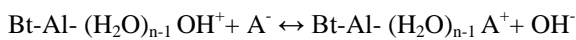
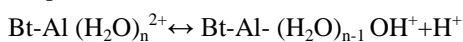
Previous studies on Al modified clay suggested that the underlying phosphate adsorption mechanism is an ion exchange mechanism with the OH functional surface group. There is limited information about the Phosphorus adsorption mechanism by chitosan lanthanum adsorbent. As shown in Figure 2, both adsorbents showed high removal efficiency at pH 5 where the removal efficiency decreased with increasing pH to 8 this can support the OH exchange mechanism. Kamble 2007 studied the adsorption of Fluoride on Chitosan Lanthanum adsorbent. The study

concluded that Fluoride adsorption could be explained in terms of ligand exchange mechanism between fluoride ion and hydroxide ion coordinated on lanthanum immobilized on chitosan. This can be simplified by the following equation (Kamble, *et al.*, 2007):



Where Ct is chitosan and A⁻ is any anion such as fluoride or phosphate. The authors believe that the phosphate adsorption onto CtLa follows the same mechanism of fluoride adsorption explained by Kamble.

Similarly, the Al modified bentonite adsorption can be explained by the following simplified exchange mechanism where the phosphate exchange the OH⁻ functional groups on the adsorbent surfaces. The ion exchange mechanism can be represented by the following simplified reaction:



Where Bt is bentonite and A⁻ refers to anions.

In general, The dependence of phosphorus adsorption on pH confirms that adsorption is due to OH group exchange mechanism that is why phosphorus adsorption in both adsorbents has been affected by pH change.

4. Conclusion:

This study concludes that chitosan Lanthanum adsorbent is a good adsorbent and can be used for removal of phosphates from water and wastewater, it is more efficient than Aluminium modified bentonite. The comparison of both adsorbents showed that the adsorption capacities for Al-Bentonite and La-chitosan were 10.71 and 17.9 mg p/gm adsorbent respectively. Kinetic studies showed that the two adsorbents were well presented by the pseudo-second order kinetics. The pseudo second order rate was found to be 0.00543, and 0.0021 for Al-Bentonite, and La-chitosan respectively. Further studies should be conducted to assess the co-ions on the removal of phosphate in water.

Table 2. Parameters of the pseudo-first-order and pseudo-second-order kinetic models for phosphate adsorption removal using the two adsorbents

Pseudo first order			Pseudo-second order			Experimental value
K1	q _e	R2	K ₂	q _e	R2	q _{e exp}
0.0093	6.73	0.985	0.002188	10.42	0.999	9.5
0.002	4.23	0.751	0.005427	7.9	0.999	7.45

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