




Synthesis of chitosan based new material for recovery of Pt (IV)

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Abstract. In this study a new type of adsorbent bead precursor was reached using chitosan and pretreated clay. The pretreated clay was produced from the clay of the "Tsogt-Ovoo" deposit in the Umnugovi province of Mongolia. It was produced by acid and heat treatment to remove its mechanical impurities. The mixtures of chitosan and pretreated clay were taken with various ratios (8:1, 8:2, and 8:3) for chemical processing to obtain a chitosan-based adsorbent bead for recovery of (Pt(IV)) from aqueous solution. It was examined that the adsorption capacity of the adsorbent bead when the chitosan and pretreated clay mass ratio 8:2 was the highest for platinum ion (Pt (IV)). The adsorption properties of Pt (IV) were studied depending on the solution pH, contacting time, temperature, initial concentration of platinum ion solution, and amount of adsorbent material. The adsorption efficiency was the highest (93.29%) when the initial concentration of the platinum ion was 50 mg/l at the following condition; pH=3, temperature 250C, time 3 hours, and the amount of the adsorbent material 0.1 g. It represents that the chitosan clay bead can be applied for the recovery of platinum (Pt (IV)) from aqueous solution.

Keywords: adsorption kinetics, adsorption thermodynamics, platinum recovery

1 Introduction

The increasing demand for the platinum group metals (PGMs) for production of catalysts and electrical equipment industry in related industries, combined with the limited resources available, has led to increasing interest in the recovery of these

important elements [1,2]. The problem that separation of these metal ions is still have in the past years, because of their complex chemistry and the overlapping properties, represents a real challenge. The conventional methods for the separation of metal ions from aqueous solution include solvent extraction, oxidation, reduction, membrane filtration, precipitation, ion-exchange and adsorption [3-5]. Among the all above methods adsorption is highly effective and economical. Chitosan is an N-deacetylated derivative of chitin, a cationic polysaccharide composed of β -D-glucosamine and acetyl-B-D- glucosamine residues with a 1, 4 linkage [6].

Chitosan has been widely studied for the sorption of metal ions. The presence of amine groups gives to the biopolymer in acidic media a cationic behavior that makes it unique among polysaccharides. These amine groups are responsible for its excellent sorption properties. Metal binding may occur on the free electronic doublet of chitosan by complexation, chelation mechanisms. Other reactive groups (for example -OH groups) may be involved in the stabilization of metal-amine bonds; however, it is commonly accepted that amine groups are responsible of the binding of metal cations in near neutral solutions [7]. Kaolin is one of the most familiar natural inorganic clays, existing in rocks copiously in crystalline structure [8]. Kaolin has outstanding properties (e.g., porosity, low-cost, thermal stability, excessive availability, and non-toxicity) in environmental applications when employed as adsorbent alone and/or within a chitosan composite, such as good adsorption capacity, high surface area, easy to functionalization its surface, high thermal stability, and environmentally friendly. Recent studies represent that chitosan and kaolinite complement each other for producing multifunctional bio composite adsorbents, which can be effectively employed for removal of various water contaminants including organic anionic dyes, biological contaminants, organic cationic dyes, and nutrients. From the environmental standpoint, utilizing kaolinite powder as a filler or surface modifier to chitosan will improve the surface area and adsorptive property of chitosan, and will reduce the cost and environmental impact of solid waste disposal [8].

The functional groups have the ability to adsorb many substances such as proteins, dyes, precious and heavy metals.

However, chitosan has unfair chemical stability because it may dissolve in acidic solution such as acetic acid (CH_3COOH), nitric acid (HNO_3) and hydrochloric acid (HCl). Hence, its application is limited and chitosan needs to be modified to improve its chemical stability and adsorption capacities. Chitosan can be physical and chemical modified in order to reduce the particle size, increase surface area and adsorption. In this work the mixtures of chitosan and pretreated clay were taken with various ratios (8:1, 8:2, and 8:3) for chemical processing to obtain a chitosan kaolin composite material for recovery of (Pt (IV)) from aqueous solution [9].

2 Experimental

2.1 Materials and Reagents

Chitosan with high molecular weight and degree of 90% was purchased from NJDULY. All the other reagents used in this work were of analytical grade. The stock solutions

were prepared by dissolving these metal salt in deionized water and were further diluted as required.

2.2 Preparation of Chitosan kaolin composite material

To prepare chitosan solution, dissolving 8 g of chitosan in 200 ml of 2 % (v/v) CH₃COOH. The solution was stirred at 500 rpm for 12 h and after that chitosan and kaolinite solution was prepared by adding 1-3 g of kaolin to the prepared solution and stirring it for 6 hours. Chitosan and kaolin solution was dropped into 1 M NaOH to produce chitosan kaolin composite beads. The chitosan kaolin composite beads were chilled for 12 h and filtered from NaOH solution. Finally, chitosan kaolin composite beads were washed with DI water and dried at 333 K.

2.3 Adsorption experiment

The adsorption experiments were performed at various solution pH, adsorbent amount, contacting time, initial concentration and temperatures. The effect of solution pH onto the adsorption of Pt (IV) mixing 0.1 g of the adsorbent in 50 ml sample having an initial concentration of 50 mg/l. The pH of the samples was corrected with 0.1 M hydrochloric acid (HCl) and 0.1 M sodium hydroxide (NaOH) to different pH values in the range of pH=3-10. The temperature and contact speed were fixed at 25⁰C and 100 rpm respectively, while the flasks were shaken for 120 min. The effect of adsorbent amount on the adsorption of Pt ion was performed first mixing range of 0.025-1g in 50 ml sample having an initial concentration of 50 ppm. The contacting time effect of adsorption was studied over a time range of 30-360 min while the effect of initial concentration was investigated at a concentration range of 10-100 ppm. Adsorption thermodynamic were conducted at temperatures of 25-55⁰C. Kinetics of adsorption was performed at a concentration range of 10-100 mg/l and time range of 30-360 min. The equilibrium adsorption capacity was determined from the mass balance equation as shown in Equation (1):

$$q_e = \frac{(C_0 - C_e)}{M} \times V \quad (1)$$

where q_e (mg/g) is the equilibrium adsorption capacity, C_0 , and C_e is the initial and equilibrium concentration (mg/l) of metal ion in solution respectively, V (ml) is the volume and m (g) is the weight of the various adsorbent. The percentage removal of metal ions by adsorbent was determined as shown in Equation (2):

$$R(\%) = \frac{(C_0 - C_e)}{C_0} \times 100 \quad (2)$$

3 Theory of evaluation of data analysis

3.1 Adsorption isotherms model

The adsorption data of metal ions onto various adsorbents were analyzed with the Langmuir and Freundlich model. The Langmuir and Freundlich equation is described

in their linear form in Equation (3) and (4). The Langmuir model is based on the fact that every adsorption site is identical and energetical equivalent. It predicts that the adsorption occurs at specific homogeneous sites on the adsorbent and this is described in monolayer adsorption processes [10].

$$\frac{C_e}{q_e} = \frac{C_e}{Q_m} + \frac{1}{Q_m b} \quad (3)$$

where: q_e is the equilibrium adsorption amounts on the beads (mg/g), C_e is the equilibrium concentration of solute ion in the bulk solution. q_m (mg/g) and b (1/mg) shows capacity and a constant related to the adsorption equilibrium constant to monolayer adsorption. The Langmuir model parameters can be estimated from the slope and intercept of the linear plot of C_e/q_e versus C_e . The empirical Freundlich isotherm is based on the equilibrium relationship between heterogeneous surfaces:

$$\log q_e = \log K_f + \frac{1}{n} \log C_e \quad (4)$$

In above equations, K_f (mg/g) and n are infinitesimal constant representing the adsorption capacity and adsorption intensity respectively. Under usual adsorption conditions, the values of n should be in the range of 1-10 [11]. The parameters of Freundlich model can be estimated from the slope and intercept of the linear plot of $\log q_e$ versus $\log C_e$.

3.2 Thermodynamic studies

Thermodynamic investigations are another most of effective parameter of adsorption studies. Adsorption temperature is shown to affect adsorption capacity. Usually adsorption capacity increased when temperature is getting higher, the process is termed endothermic, and when adsorption decreases with temperature the process is called exothermic. The thermodynamic parameters present the essential parameters for subsequent engineering evaluation on the ultimate uptake of the adsorbents, and, hopefully, also provide insights to the adsorption mechanisms thus applied for further use in process modification and optimization [12]. The entropy and enthalpy change, associated with the process can be estimated from Eq. (5):

$$\ln K = -\frac{\Delta H^0}{RT} + \frac{\Delta S^0}{R} \quad (5)$$

The changes of Gibbs free energy ΔG^0 is the fundamental criterion of spontaneity. If ΔG^0 is a negative, reactions occur spontaneously but ΔG^0 is a positive, reactions occur non spontaneous reaction at a given temperature [13]. The free energy of the adsorption reaction, calculating the adsorption equilibrium constant, K , is given by Eq. (6):

$$\Delta G^0 = -RT \ln K \quad (6)$$

The equilibrium constant K as stabled mathematically by Liu et al. [11] is given in Eq. (7):

$$K = \frac{q_e}{C_e} \quad (7)$$

where q_e (mg/g) is amount of platinum ions adsorbed on the adsorbent and C_e (mg/l) is the equilibrium concentration. The gas constant R is defined by 8.3145 J/mol K; T ($^{\circ}\text{C}$) is the temperature of the solution; ΔH^0 is the enthalpy change, ΔS^0 is the entropy change. ΔH^0 and ΔS^0 can be estimated from the slope and intercept of a plot of function of $1/T$ as a LnK .

3.3 Kinetic models

The adsorption kinetics provides insight into the reaction rate and the sorption mechanism involving mass transfer, diffusion, and reaction on the adsorbent surface during adsorption. The process of adsorption of adsorbates from aqueous solution. It is also important to determine whether the behavior of metal ion uptake can be explained by a predictive model. The adsorption data of metal ions onto was described with respect to the Lagergren pseudo-first-order kinetic model [14], and pseudo-second-order model that was presented by Ho and McKay [15]. The models are shown in Eq. (8) and (9):

$$\log(q_e - q_t) = \log(q_e) - \frac{K_1}{2.303} t \quad (8)$$

Where q_e and q_t (mg/g) shows the amount of platinum ions absorbed on the adsorbent at equilibrium and time t respectively. K_1 (min^{-1}) is the rate constant of the pseudo-first order kinetics. The value of adsorption rate constant, K_1 , can be estimated from the straight-line plot of t versus $\log(q_e - q_t)$:

$$\frac{t}{q_t} = \frac{1}{K_2 q_e^2} + \frac{1}{q_e} t \quad (9)$$

where K_2 (g/mg min^{-1}) is the rate constant for a pseudo-second order model and the definitions of q_e and q_t remains the same. The slope and intercept of the linear plot of t/q_t versus t , gives the values of q_e and K_2 respectively.

4 Results and discussion

4.1 Effect of pH

The Pt (IV) adsorption onto chitosan beads is strongly influenced by acidity (pH), as presented in Fig. 1 This is due to the existence of $-\text{NH}_2$, and $-\text{OH}$ active groups in chitosan beads which could be covalently bonded with the other substance on certain conditions. Chitosan beads can electrostatically interact with other negatively charged substances.

4.2 Effect of contacting time, initial concentration and kinetic studies

Fig. 2 presented the effect of contacting time on the Pt (IV) adsorption by chitosan kaolin beads. The results shows which the adsorption increases with increase in contacting time and attained the equilibrium at around 3 hours. It is very clear from these results that the contacting time is required for maximum adsorption of metal ions by chitosan kaolin beads was dependent on the initial metal ion concentration. Based on these results, the contact time was fixed about 3 hours for rest of the batch

experiments. Experimental studies were carried out with varying initial metal ion concentrations of Pt (IV) ranging from 10 to 100 mg/l using 0.1 g of adsorbent dose at pH=3.0 for Pt (IV). The results shows that the adsorbed Pt (IV) amount increases with increasing metal ion initial concentration and also the adsorption percentage decreases with increasing initial metal ion concentration. This is due to the total available sites are limited at fixed adsorbent dose, thereby adsorbing almost the same amount of solute, resulting in a decreasing of percentage adsorption corresponding to an increase in initial metal ion concentration.

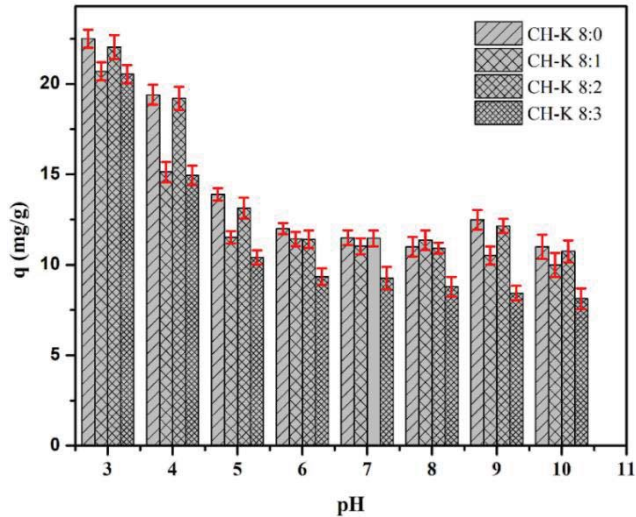


Figure 1. pH effect on Pt (IV) adsorption. (Conditions: initial metal ion concentration 50 mg/l, contacting time 2h, adsorption amount 0.1 g)

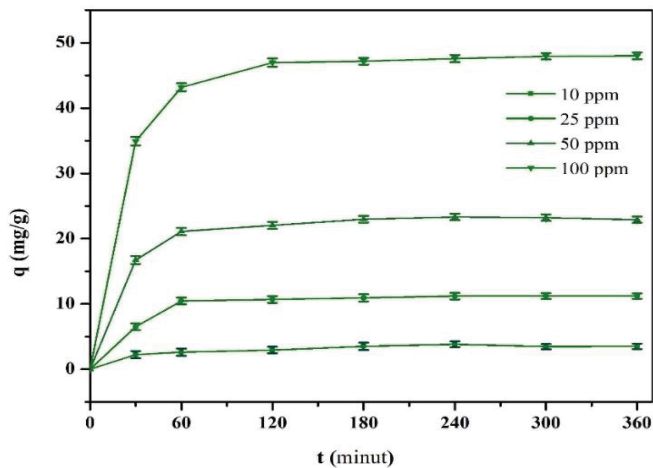


Figure 2. Contacting time effect on the Pt (IV) adsorption of various initial concentration. (Conditions: concentrations 10, 25, 50 and 100 mg/l, solution pH=3.0, adsorbent amount 0.1 g)

Table 1. Kinetic parameters for the adsorption of platinum ion onto CH-K 8:2 beads at pH=3

T(K)	C_0 (mg/l)	q_e (exp)	First- order kinetic model			Second-order kinetic model		
			K_1 (l/min)	q_e (cal) (mg/g)	R^2	K_2 (g/mg/min ⁻¹)	q_e (cal) (mg/g)	R^2
298	10	3.512	0.211	8.426	0.784	0.683	3.761	0.997
	25	11.233	0.114	14.333	0.904	0.164	12.469	0.986
	50	23.333	0.095	19.106	0.899	0.286	23.753	0.999
	100	48.022	0.095	30.177	0.962	0.032	52.356	0.992

The result obtained from the experiment carried were used to investigate the kinetics of platinum ion adsorption onto chitosan kaolin beads. The pseudo-first-order and pseudo-second-order model was applied to fit the kinetic data. Recent studies have displayed that the pseudo-second-order kinetic model gives a better fit for adsorption data [10]. Table 1 gives the values of the parameters for pseudo-first-order and second-order kinetic model, and it was conformed that the correlation coefficients for the straight plot of t/q_t against t from the pseudo-second-order rate law gave the best fit for Pt (IV) ion in contrast to the correlation coefficient of the pseudo-first-order kinetic model obtained from the linear plot of $\log(q_e - q_t)$ against t . To further investigate kinetic data the However, there was a large difference for q_e values obtained by calculating from pseudo-first-order kinetic model and experimental values. In the case of the pseudo-second-order model, the q_e (cal) values agree very well with the experimental data. This provides that the adsorption of platinum ion onto chitosan kaolin beads is a pseudo-second-order reaction model. Pseudo-second-order reaction model is based on the prediction that the rate limiting step may be chemical adsorption or chemisorption involving valence forces through exchange of electrons between adsorbate (metal ions) and adsorbent (chitosan kaolin beads), provides best correlation of data [16].

4.3 Temperature of thermodynamic parameters

The temperature effect on Pt (IV) ion adsorption by chitosan kaolin adsorbent bead was studied at 25, 35, 45 and 55°C, and initial metal concentration was 50 mg/l. The results found that the adsorption capacity increased with temperature is getting higher. This resulted that the adsorption process is endothermic process in this condition. The thermodynamic parameters are shown in Table 2. The ΔG^0 values are negative, it means that adsorption was spontaneous. As the temperature increases, the ΔG^0 values decrease, indicating less driving force and hence resulting lesser adsorption capacity at higher temperatures. The values of ΔH^0 and ΔS^0 are positive, it suggested that adsorption process was endothermic nature and the determined the disorderliness of the sorption at the solid-liquid interface. It was then suggested that the adsorption of Pt (IV) on chitosan kaolin bead could be mainly dominated by chemisorption.

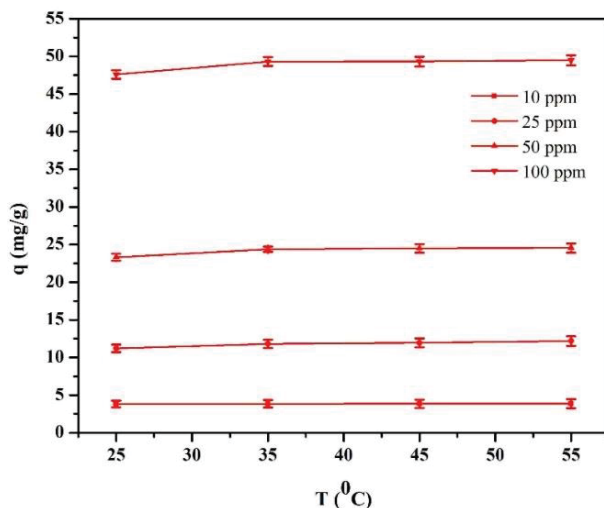


Figure 3. Temperature effect on the Pt (IV) adsorption by CH-K 8:2 beads. (Conditions: initial metal ion concentrations 10, 25, 50 and 100 mg/l, solution pH=3.0, adsorbent amount 0.1 g, contacting time 3 hours)

Table 2. Thermodynamic parameters for the adsorption of platinum ion onto CH-K 8:2 beads at an initial concentration of 50 mg/l.

T (K)	C_0 , mg/l	ΔG^0 , kJ/mol	ΔH^0 , kJ/mol	ΔS^0 , kJ/mol·K	R^2
298	50	-4.805	18.705	79.234	0.95
308		-5.786			
318		-6.647			
328		-7.138			

4.4 Adsorption isotherm parameters

The Langmuir and Freundlich model were used to determine the appropriate isotherm for platinum onto chitosan kaolin composite adsorbent bead. Adsorption isotherms explain the interactive process between the adsorbents and adsorbates in aqueous medium at the attained saturation point. Adsorption isotherms of Pt (IV) on chitosan kaolin bead were identified with different initial concentrations from 10 to 100 mg/L under optimized conditions in terms of the pH=3, contacting time (3 hours), and amount of the adsorbent 0.1g. Typical adsorption isotherms, the Langmuir and Freundlich models were applied to evaluate the adsorption of Pt by chitosan kaolin bead (Fig. 4, 5). The closer R^2 is to one the best the model fit. Langmuir isotherm can be used to interpret the type of sorption using the Hall separation factor (K_L), which is favorable ($0 < K_L < 1$), unfavorable ($K_L < 0$), linear ($K_L = 1$) or irreversible ($K_L = 0$). As shown in Table 3 and Fig. 4 and 5. The R^2 value in the Langmuir isotherm was larger than that in the Freundlich isotherm. The maximum adsorption capacity (q_{max}) estimated from the

Langmuir model was 314 mg/g, which suggested that the adsorption of Pt (IV) on chitosan kaolin bead mainly occurred by a monolayer reaction.

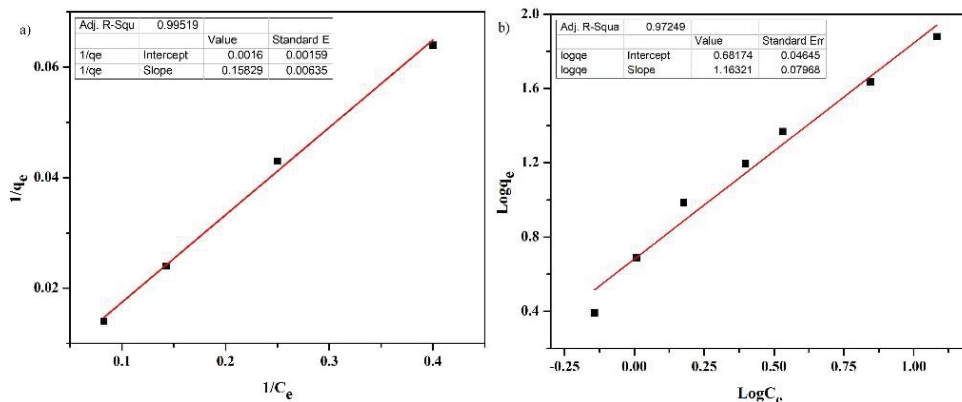


Figure 4. a) Langmuir isotherm model, b) Freundlich isotherm model

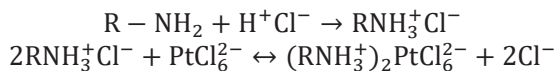
Table 3. Langmuir and Freundlich isotherm parameters for Pt (IV)

Metal ion	Isotherm model	Parameters	Pt (IV)
Pt (IV)	Langmuir	q_{max} , mg/g	314
		b , l/mg	0.02
		K_L	0.01
	Freundlich	R^2	0.99
		n	0.86
		K_f	37.13
		R^2	0.97

5 Discussion

Different pH values (pH 3, 4, 5, 6, 7, 8, 9 and 10) have been examined to determine the optimum pH in the adsorption of Pt (IV) within 3 h of contacting time. As shown in Fig 1, the optimum adsorption of Pt (IV) is achieved at pH=3. At pH=4-10, the adsorption capacity value decreases. The speciation of Pt (IV) depends on both pH and chloride concentrations. At low pH, platinum is usually present in solution in stable form Pt (IV).

It can form stable complexes especially with amino group chelation sites of chitosan kaolin composite bead. Pt (IV) being provided as K_2PtCl_6 in this study, the amount of chloride in the solution is high enough to favor the formation of chloro-anionic species ($PtCl_n^{(n-4)-}$) that will be adsorbed on protonated amine groups of chitosan kaolin composite bead. The protonation of amine groups on the chitosan kaolin composite bead reacted an electrostatic attraction of anionic metal complexes and increased the number of available binding sites for platinum ion uptake. In the presence of chloride ions the interaction between metal anions and active sites of the chitosan kaolin beads are shown below.



6 Conclusion

The adsorption of Pt (IV) was investigated in this study by chitosan kaolin composite beads. The results approved that the adsorption process is dependent on contact time, initial metal ion concentration, adsorbent amount, solution of pH and temperature and contacting time. The adsorption capacity of 8:2 chitosan kaolin composite bead is higher than that other composite and pure bead. The maximum adsorption capacity was obtained at pH=3 for Pt (IV). Adsorption kinetic data were fitted well by the pseudo-second-order model, and the adsorption isotherm data were described well by the Freundlich model. The maximum adsorption capacity for Pt (IV) was found to be 314.0 mg/g. The thermodynamic calculation demonstrates spontaneous and endothermic nature of Pt (IV) onto chitosan kaolin composite beads.

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