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RESEARCH ARTICLE

ADSORPTION OF NITRATE IONS ONTO CAMEROONIAN'S VOLCANIC SOILS: KINETICS AND EQUILIBRIUM STUDIES

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ABSTRACT

The retention capacity of nitrate ions by adsorption onto two volcanic soils collected from the localities of Tiko (S-TIK) and Tombel (S-BEL) in the South-west region of Cameroon was investigated. The influence of parameters such as, contact time, pH, adsorbent dose and initial concentration of the nitrate ions were studied. The experimental results obtained show that the amount of nitrate ions adsorbed increased with contact time and reached equilibrium at 45 min and 50 min respectively for S-BEL and S-TIK. The optimal pH of adsorption of the nitrate ions is 2.0 for both adsorbent with maximum adsorption of 3.85 mg/g for S-BEL and 4.32 mg/g for S-TIK. The isotherm models used to characterize the adsorption processes were Langmuir, Freundlich, Tempkin and Dubinin-Kaganer-Raduskevich (D.K.R). The Freundlich and Tempkin models better describe the adsorption process with S-BEL while Tempkin and D.K.R models better describe the adsorption process with S-TIK. The kinetic study was carried out using pseudo first order, pseudo second order, Elovich and intraparticle diffusion. It was found that the adsorption fitted the Elovich and Intraparticle diffusion models for both adsorbents. These results showed that the retention capacity of nitrate ions is greater on S-TIK compared to S-BEL.

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INTRODUCTION

The water quality still remains characterized by some problems of qualitative and quantitative degradation of their resources, to which it is added the proliferation of the centre of pollution (Nabila *et al.*, 2011). The centre of pollution can be of diffused origin, coming mainly from industrial and agricultural activities. They can also be issued from domestic waste waters and organic industries like the manufacture of explosives (J.J. Anguile *et al.*, 2013). All natural water contains nitrates with different concentration with respect to the season. Generally, in the nitrogen's cycle their concentration in surface water does not exceed the range of 3.0 to 5.0 mg/L with only some mg/L in subsoil waters (F. Balducchi, 1994). Unlike other pollutants, nitrates ions are not toxic in themselves, but their transformation by the micro-organisms into nitrites and nitrous compounds cause medical disorders on human being and contribute to the pollution of the ecosystems (F. Balducchi, 1994). Taking into account the extent of pollution, the maximum nitrates ions rate in drinking water is fixed at 50 mg/L by the World Health Organization. The increase of nitrates ions content also has a defavorable environmental

impact owing to the massive proliferation of green alga which contributes to the acceleration of global warming and to eutrophication (Nizar Aouina, 2009). When the concentration of the nitrate ions in the environment exceeds the permissible amount, the process of depollution becomes urgent and constitutes a challenge. The electrochemical methods employed for elimination of nitrate ions include the catalytic processes of reduction, osmosis, nanofiltration, electrolysis, precipitation, ionic exchange and electro dialysis (C. Namasivayam, 2005). These methods are very expensive and require high energy consumption, thus it is necessary to seek for techniques that are low cost and effective such as the adsorption process (Ketcha *et al.*, 2011). In the last decade, many adsorbents have been used such as activated carbon (C. Namasivayam *et al.* 2005), iron oxides (N. Ozturket *al.* 2004), red soil (R. Shashikantet *al.* 2013) and clays (B. Nabila *et al.* 2011). In this present work, the emphasis has been put on volcanic soils because of their specific properties such as a high percentage of organic carbon, a strong holding capacity, a low apparent density and a wealth of exchangeable cation which is the dominant property (P. Legrand *et al.*, 2007). Thus, the aim of this study was to evaluate the retention capacity of the nitrate ions onto two volcanic soils namely S-TIK and S-BEL.

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MATERIALS AND METHODS

Adsorbents preparation

The volcanic soil was collected precisely in the localities of Tiko and Tombel in the South-west region of Cameroon. 20.0 g of soil was taken and mixed with 75.0 mL of 2.0 M potassium iodine solution and vigorously stirred during 1.0 hour, then filtered in order to remove existing nitrate ions. The solid residue obtained was dried during 24 hours then was crushed in a mortar. Finally, the powder obtained was used as adsorbent throughout the experiments.

Adsorbate preparation

The nitrate ions stock solution of concentration 1000 mg/L was prepared by dissolving (1.6400±0.0001) g of KNO₃ in 1.0 L volumetric flask as described by Akosman *et al* (2010). The lesser concentrations of nitrate ions were obtained with dilution process.

Salicylic acid solution preparation

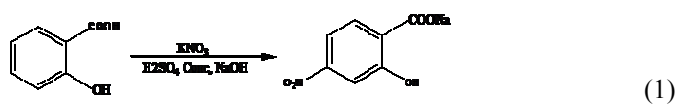
The solution was obtained by dissolving 5.0 g of salicylic acid in 100.0 mL of concentrated sulphuric acid. The preparation was made seven days before the use and it is stable when it is stored safe from the light and in a fresh place (D. A. Cataldo, 1975).

Batch adsorption experiments

Batch adsorption was carried out at room temperature. The effect of contact time, adsorbent dose, pH of solution and the initial concentration of nitrate ions were studied. For each experiment, 20.0 mL of nitrate ions solution of known concentration was mixed with a known mass of adsorbent. The pH of solution was adjusted either with 0.1 N HCl or 0.1 N NaOH. The concentration of nitrate ions after was determined by using colorimetric method at wave length of 410 nm using a UV-Visible spectrophotometer Techmel S23A at the maximum absorption wavelength of 1000nm, as described by Namasivayam *et al* (2005).

Adsorption equilibrium studies

Batch adsorption tests were carried out by adding a fixed amount of adsorbent (0.1 g) into 250 mL conical flasks containing 20 mL of KNO₃ for different initial concentrations (20-100mg/L) at room temperature and at pH of 2. The flasks were placed on a shaker and suspension was stirred for 45 minutes with S-BEL and 50 minutes with S-TIK by using a magnetic stirrer. After filtration, the solution obtained is complex according to equation 1.



To form the complex above, we used method described by Namasivayam *et al* (2005). The amount of adsorption at equilibrium, Q_e (mg/g) was calculated by:

$$Q_e = \frac{(C_0 - C_e)}{m} * V \quad (2)$$

Where, C_0 and C_e (mg/L) are the liquid-phase concentrations of nitrate ions at initial and equilibrium, respectively. V is the volume of the solution (L) and m is the mass of dry adsorbent used (g). The equilibrium data were then fitted by using Langmuir, Freundlich, Tempkin and Dubinin-Kaganer-Radushkevich adsorption isotherm models.

Langmuir adsorption isotherm

The Langmuir adsorption isotherm is used for adsorption of a solute from a liquid solution. The Langmuir adsorption isotherm is often expressed as:

$$\frac{Q_e}{Q_m} = \frac{KC_e}{1 + KC_e} \quad (3)$$

Where, Q_e (mg/g) is the adsorption density at the equilibrium, c_e is the equilibrium concentration of adsorbate in solution (mg/L), Q_m (mg/g) is the maximum adsorption capacity corresponding to complete monolayer coverage. K (L/g) is the Langmuir constant, and it is related to energy of adsorption. The linear form of this equation is as follows:

$$\frac{1}{Q_e} = \frac{1}{Q_m} + \frac{1}{K \cdot Q_m} * \frac{1}{C_e} \quad (4)$$

Freundlich adsorption isotherm studies

Freundlich equation is an equation based on the adsorption onto a heterogeneous surface (J.M. Ketcha *et al.*, 2012). It is often expressed as the Equation 5 below:

$$Q_e = K_f C_e^{1/n} \quad (5)$$

Where Q_e (mg/g) is the quantity of solute adsorbed at equilibrium, C_e (mg/L) is the concentration of adsorbate at equilibrium, K_f and n are empirical constants dependent on several factors. This equation is conveniently used in a linear form by taking the logarithms of both sides as:

$$\ln Q_e = \ln K_f + \frac{1}{n} \ln C_e \quad (6)$$

Tempkin adsorption isotherm

The Tempkin isotherm supposes that the heat of adsorption decreases linearly with the insurance due to the adsorbent. It is generally expressed in the following form:

$$Q_e = \frac{RT}{b_T} (\ln A_T + \ln C_e) \quad (7)$$

Where A_T is the Tempkin's constant (L/g), B_T a constant depending on the heat of sorption, R is constant of perfect gases (8,314 J.mol⁻¹.K⁻¹), T is absolute temperature (K) and Q_e is quantity adsorbed with balance (mg/g).

Dubinin-Kaganer-Radushkevich adsorption isotherm

Langmuir and Freundlich isotherms are insufficient to explain the physical and chemical characteristics of adsorption. It is why the D-K-R isotherm is commonly used to describe the sorption isotherms of single solute systems.

The D-K-R isotherm, apart from being analogue of Langmuir isotherm, is more general than Langmuir isotherm as it rejects the homogeneous surface or constant adsorption potential. The D-K-R isotherm is expressed as:

$$Q_e = Q_{max} \exp \left[\frac{(RT \ln(1 + \frac{1}{C_e}))^2}{-2Ea^2} \right] \quad (8)$$

Where, E_a (KJ/mol) is the main energy of adsorption and gives information about the physical and chemical features of adsorption. The linear form of the D-K-R isotherm equation is expressed below:

$$\ln Q_e = \ln Q_{max} - \beta \epsilon^2 \quad (9)$$

Where, $\epsilon = RT \ln(1 + \frac{1}{C_e})$ is called Polanyi potential.

Kinetic adsorption studies

In the literature, several models have been applied to know order of the adsorbent-adsorbate interactions and the rate of adsorption process. In this study, four kinetic models have been applied confronted to the experimental data.

The pseudo first order model

This model assumes that the speed of sorption at the moment, t is proportional to the difference between the quantities adsorbed with balance Q_e and the quantity adsorbed at this moment Q_t . In this model, adsorption is reversible (R. Calvert, 2003). The rate constant of adsorption is expressed as a first order rate expression (S. Lagergren, 1898) as shown below:

$$\frac{dQ_t}{dt} = k_1(Q_e - Q_t) \quad (10)$$

Where, Q_e and Q_t are the sorption capacity at equilibrium and at time t respectively (mg/g) and k_1 is the rate constant of pseudo-first order sorption (L/min). After integration and applying boundary conditions, $t = 0$ to

$t = t$ and $Q_t=0$ to $Q_t=Q_t$, the integrated form of equation is:

$$\ln(Q_e - Q_t) = k_1 t \quad (11)$$

The pseudo second order model

Described by Ho et al (2000), this pseudo second order model makes it possible to describe the kinetics of the reaction of fixing with pollutants on the adsorbent. It results in the relation:

$$\frac{dQ_t}{dt} = k_2(Q_e - Q_t)^2 \quad (12)$$

Where Q_e and Q_t are the adsorption capacity at equilibrium and at time t , respectively (mg/g) and k_2 is the rate constant for the pseudo-second order adsorption (g/mg.min). For boundary conditions, $t=0$ to $t=t$ and $Q_t=0$ to $Q_t=Q_e$, the integrated and rearranged form of Equation 13 is:

$$\frac{t}{Q_t} = \frac{1}{k_2 Q_e^2} + \frac{t}{Q_e} \quad (13)$$

Elovich equation

The Elovich equation is generally expressed as Chien et al., (1980):

$$\frac{dQ_t}{dt} = \alpha \exp(-\beta Q_t) \quad (14)$$

Where Q_t is the sorption capacity at time t (mg/g), α is the initial adsorption rate (mg/(g)(min)), and β is the desorption constant (mg/(g)(min)) during any one experiment. The integrated and simplified equation (assuming that $\alpha\beta t \gg 1$) is:

$$Q_t = \frac{1}{\beta} \ln(\alpha\beta) + \frac{1}{\beta} \ln(t) \quad (15)$$

The intraparticle diffusion model

The intra-particulate diffusion model proposed by Weber and Morris (F.C. Wu et al., 2001) the equation is:

$$R = k_{int} t^\alpha \quad (16)$$

Where R is the percentage of nitrate ions adsorbed, t is the contact time, α is a constant whose value depends on the adsorption mechanism and K_{int} is the intraparticle diffusion rate constant (1/min). A linear form of equation 17 is:

$$\ln R = \ln k_{int} + \alpha \ln t \quad (17)$$

RESULTS AND DISCUSSION

Chemical characterization

The Table 1 below presents the physicochemical characteristics of adsorbents (S-BEL and S-TIK) used. From the spectra recorded on KBr disc method, it can be observed the following absorption bands:

Table 1 Physicochemical characteristics of S-BEL and S-TIK

Characteristics of the sites of taking away		
Sampling Sites	Tombel	Tiko
Horizon	A	A
Depth (cm)	13.00	15.00
Chemical composition		
Moisture (%)	16.32	8.60
Organic carbon (%)	4.55	4.34
Total Nitrogen (%)	0.20	0.31
Phosphorus available (mg/kg)	80.00	151.00
pH of water solution	5.64	5.68
pH of KCl solution	4.67	5.00
K^+ exchangeable (cmol/kg)	0.78	0.75
Na^+ exchangeable (cmol/kg)	0.10	0.09
Mg^{2+} exchangeable (cmol/kg)	0.88	0.96
Ca^{2+} exchangeable (cmol/kg)	7.17	1.25
Exchangeable acidity (cmol/kg)	0.04	0.06
Cation exchange capacity (cmol/kg)	28.30	7.12

According to the FTIR spectrum of S-BEL and S-TIK (Figure 1), the broad band at 3623 cm^{-1} for S-BEL; between 3693 cm^{-1} and 3618 cm^{-1} for S-TIK corresponding to O-H vibration of elongation being able to come from water on the surface of the two soils.

Table 2. Summary of the peaks IR of S-BEL and S-TIK before adsorption

S-BEL (cm ⁻¹)	S-TIK (cm ⁻¹)	Attribution	Literature value (cm ⁻¹)
3623	3693 ; 3618	-OH	3648 (C. Akosman <i>etal</i> (2010)
3338	3291	Al-OH ou Si-OH	3325 (J.J Anguile <i>et al</i> , 2013)
1001 ; 909	999 ; 909	Si-O	1114 (J.J Anguile <i>et al</i> , 2013)
793 ; 745	741	Al-O	748 (D.N. Dinka' a <i>et al</i> , 2012)
522	459	Si-O-Al	518 (J.J. Anguile <i>et al</i> , 2013)
417	450	Si-O-Si	414 (C. Akosman <i>etal</i> ,2010)

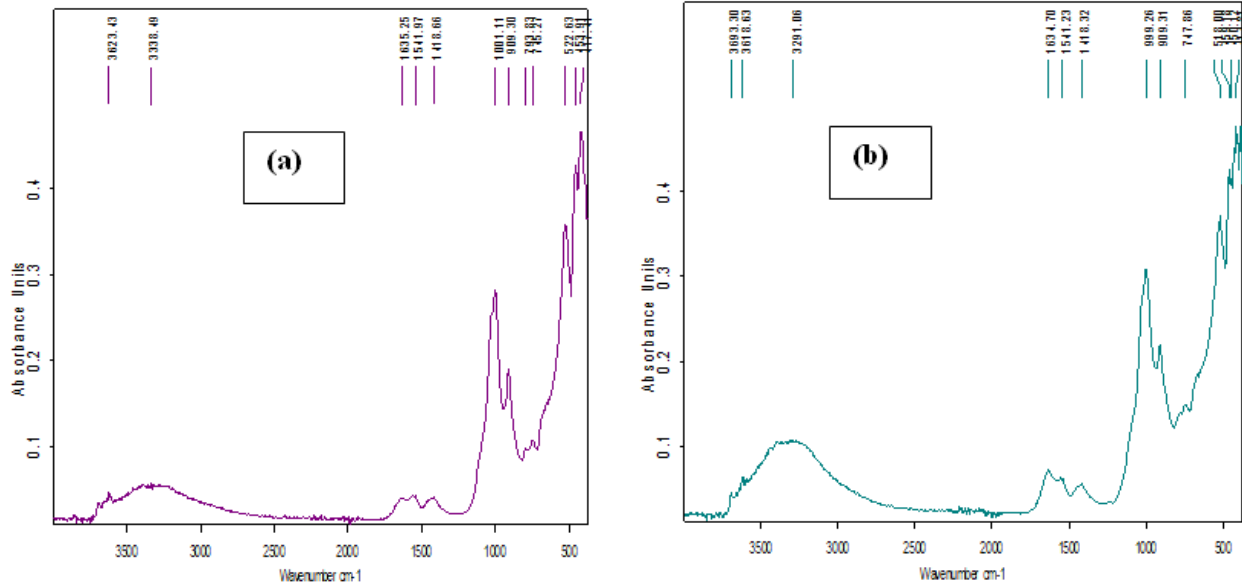


Figure 1. FTIR of S-BEL (a) and S-TIK (b)

The peaks observed at 3338 cm⁻¹ and 3291 cm⁻¹ corresponding to Al-OH yet Si-OH vibrations of deformation. These spectra reveal also the presence of the various bonds Si-O; Al-O; Si-O-Al; Si-O-Si on the surface of the two materials.

Effect of contact time

The Figure 2 below shows kinetics of adsorption of nitrate ions which reach equilibrium time after 45 min and 50 min with adsorbed maximum quantities of 3.533 mg/g and 4.133 mg/g for S-BEL and S-TIK respectively. Two different phases are observed clearly: The first phase is faster within 10 minutes for S-TIK and 30 minutes for S-BEL, consequence of the available and free adsorption sites on the surface of the adsorbents.

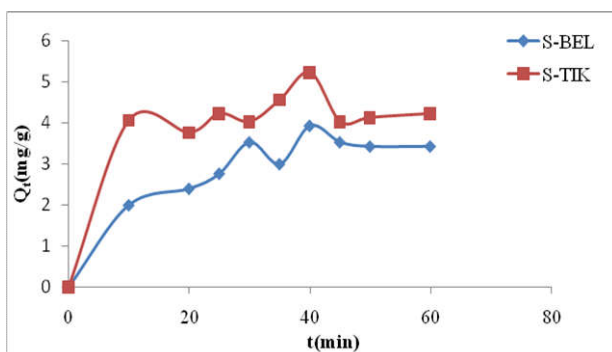


Figure 2. Influence of contact time on the adsorption of the nitrate ions

After this phase, the adsorbed quantities decrease for S-BEL and vary little for S-TIK until equilibrium, it is the slow phase because once the nitrate ions in solution are fixed at the surface of the adsorbents, thus, the pores are blocked and preventing remaining nitrate ions in solution to fix themselves. It can be noticed that the adsorbed quantities on S-BEL are lower than those on S-TIK and it can be concluded that the nitrate ions are more mobile on S-BEL compared to S-TIK.

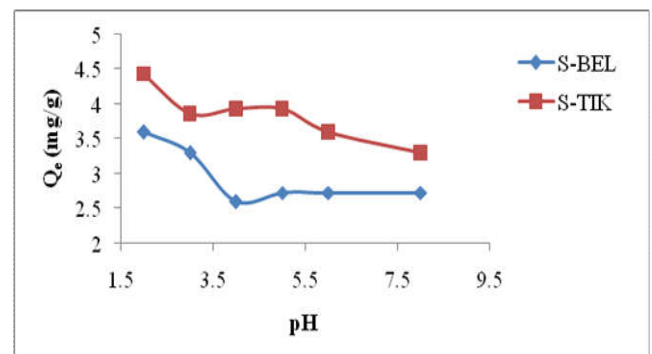


Figure 3: Effect of the pH on the adsorption of the nitrate ions

Effect of pH

The Figure 3 below shows that the pH is a significant factor in the adsorption phenomenon. Indeed, the adsorbed quantities are maximum at pH=2 and are equal to 3.6 and 4.433 mg/g respectively for S-BEL and S-TIK. This could be due to the influence of the cation H⁺ on micelle of the soil which are at the origin of the strong significant electrostatic attractions between the surface of the adsorbents and the nitrate ions.

At the $pH > 2$, the adsorbed quantity decreases as the pH increases and this is the consequence to the reduction in the degree of protonation of the surface of adsorbents (G. Fumba and *al*, 2014). The small adsorbed quantity at $pH=8$ can be justified by the competition between the ions O^{H^-} and ions $N O_3^-$. Moreover, at this pH , the ions O^{H^-} are more mobile than ions $N O_3^-$ because of their low molecular weight. Thus, the adsorbent becomes negatively charged causing an electrostatic repulsion of the negative charges on the surface of the adsorbent. This means that adsorption is not possible by ionic exchange mechanism in the basic medium but rather by the chemisorption mechanism as described by Namasivayam *et al* (2005).

Effect of adsorbate dose

The Figure 4 reveals that the adsorbed quantities decrease with an increase of adsorbent dose. This can be explained by the formation of aggregates on the surface of the adsorbents which block the access to pores. In consequence, favorite the desorbing of ions nitrate in the narrow sites and contributes to increase in the diffusion process. Similar trend was observed by Fumba and *al* (2014).

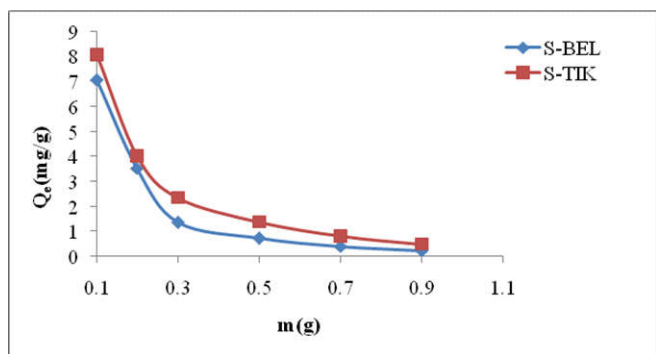


Figure 4. Influence mass of adsorbent on the adsorbed quantity

Effect of initial concentration

The Figure 5 below represents an increase in the quantities adsorbed from 1.200 to 15.567 and 1.387 to 16.360 mg/g for S-BEL and S-TIK respectively as the initial concentration of $N O_3^-$ ions increase. This progression is due to the fact that as the nitrate ions concentration increases the collision between the nitrates ions also raised on the adsorption sites (K. Bhaltachouya *et al.*,2006).

Isotherm study

The adsorption isotherm of nitrates ions on S-BEL and S-TIK is given by Figure 5. According to that graph, is an isotherm of type V. Therefore, the adsorbents used are seemed to be mesoporous and that, there are weak interactions between the S-BEL and S-TIK with respect to the nitrates ions. The presence of two-stages can result in the formation of two successive layers of nitrates ions on the surface of S-BEL and S-TIK at the time of the interaction between the nitrates ions and each adsorbent. Thus the sites of adsorption of the second layer will start to be occupied when those of the first layer are completely saturated. *Kinetic of adsorption*

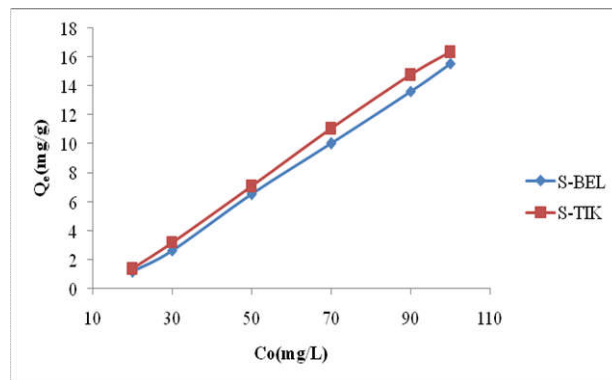


Figure 5. Influence initial concentration of the adsorbate according to the adsorbed quantity

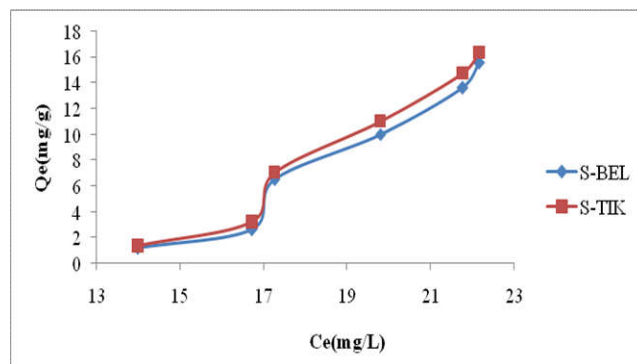


Figure 6. Influence of concentration with the balances adsorbate according to the adsorbed quantities

Table 3. Constants speed and coefficients of correlation of the kinetic models

Isotherms	Constants	S-BEL	S-TIK
Pseudo premier order	R ²	0.595	0.060
	K ₁ (min ⁻¹)	0.004	0.001
Pseudo second order	R ²	0.945	0.974
	K ₂ (g.min ⁻¹ .mg ⁻¹)	0.043	0.590
	Q _e (mg.g ⁻¹)	3.850	4.317
Elovich	R ²	0.976	0.166
	α x 10 ⁸ (mg/g.min)	0.822	8.653
Intraparticule diffusion	β (g/mg)	1.164	6.181
	R ²	0.960	0.110
	A	0.335	0.055
	K _{id} (mg/g.min ^{1/2})	9.197	37.226

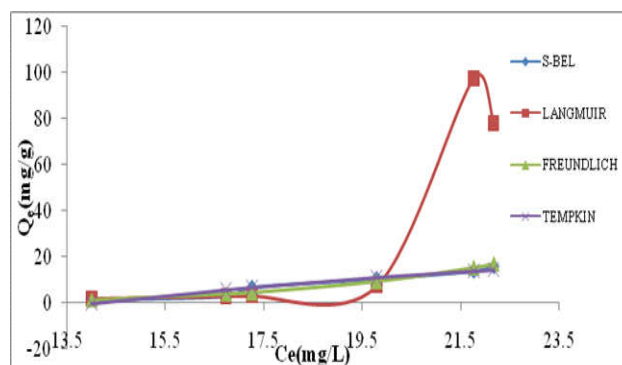


Figure 7: Modeling of Langmuir, Freundlich and Tempkin model on S-BEL

It is clearly arises from the analysis of the data that, the pseudo second order model better describes the adsorption on the surface of S-BEL and S-TIK.

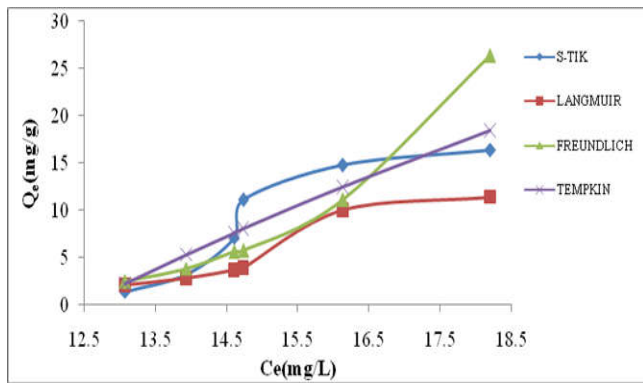


Figure 8. Modeling of Langmuir, Freundlich and Tempkin model on S-TIK

Table 4: Constant of Langmuir, Freundlich, Tempkin and D-K-R

Isotherms		S-BEL	S-TIK
Langmuir	R ²	0.865	0.619
	R ²	0.938	0.753
Freundlich	$K_F \times 10^7 ((\text{mg/g})(\text{L/mg})^{1/n})$	5.812	0.223
	1/n	5.544	7.201
	R ²	0.931	0.873
Tempkin	B (J/mol)	77.830	50.670
	A (l/g)	0.071	0.079
D-K-R	R ²	0.981	0.828
	$K' \times 10^{-4}$	0.300	2.000
	Q _m (mg/g)	29.880	416.960
	E (KJ/mol)	129.099	50.000

This implies that the adsorption of nitrate ions on the adsorbent could be governed by the chemical process (D.N. Dinka' *et al.*,2012; J.S. Essomba *et al.*,2014). The high values of the correlation coefficient of Elovich model for S-BEL indicates that adsorption is done on multilayers which is in agreement with the isotherm of the type V obtained above (Figure 5). The adsorption rate of nitrates ions on S-TIK is higher than that on S-BEL. It can be concluded that during the adsorption process, chemisorption is not the limiting step on S-BEL but rather the phenomenon of diffusion and that the limiting step on S-TIK could be chemisorption. Anagho *et al* (2013) arrived to the same conclusion. The higher value of the correlation coefficient of the intraparticle diffusion model for S-BEL proves that the adsorption process is controlled by the diffusion into pore. Whereas, the low value of diffusion constants ($K_{id} < 50 \text{ mg/g.min}^{1/2}$) show that the diffusion of nitrate ions is carried out slowly on S-BEL and S-TIK, which implies the formation of the strong interactions between nitrate ions and the adsorbents. However, the correlation coefficient of the pseudo second order model is higher than that of the intraparticle diffusion model on S-TIK thus the mechanism of adsorption is controlled by chemisorption. These results are similar to the observations done by Belaid *et al* (2010) who did the kinetic and thermodynamic studies of adsorption of dyes.

Adsorption isotherm

The analysis of the experimental data conducted to conclude that the model of D-K-R and Tempkin are appropriate to better describe the adsorption equilibrium on S-BEL and S-TIK. On their work on the adsorption of nitrates ions on various materials, Öztürk and Bektas (2004) arrived to similar observations. The values of 1/n of Freundlich for S-BEL and S-TIK are ranged between 5.0 and 8.0 which imply that the interactions between the adsorbent and the adsorbed particles

are of the Van der Waals type (S.G. Anagho *et al.*,2013). Additionally, the values of 1/n being higher than unity, it can be said that the surface of the adsorbents are homogeneous and that adsorption can occur on monolayer with possibility of formation of other layers. The positive value of the energy of sorption of the model of Tempkin for S-BEL and S-TIK indicates a weak gravitational interaction with the adsorbate and endothermic adsorption mechanism. This value being lower than 8 kJ/mol, it can be suppose that the adsorption isotherm is of type V. The mechanism of adsorption is the ionic exchange and the interaction between S-BEL and S-TIK with nitrate ions are weak (G.M.Tagne *et al* 2013). The D-K-R model determines whether adsorption is physical or chemical. Thus energies of adsorption of the S-BEL and S-TIK being higher than 40 kJ/mol ($50 < E < 130$), this leads to a chemisorption process with possibility of formation of multilayers (S.G. Anagho *et al* 2013).

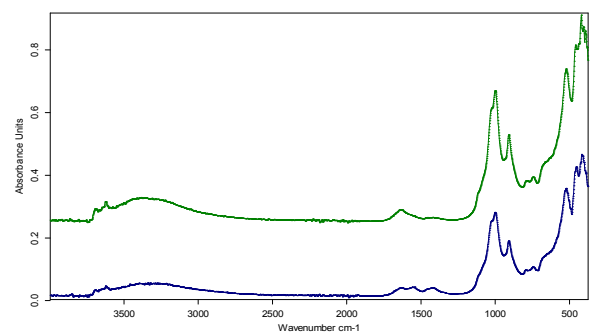


Figure 9. IR spectrum of S-BEL before and after adsorption

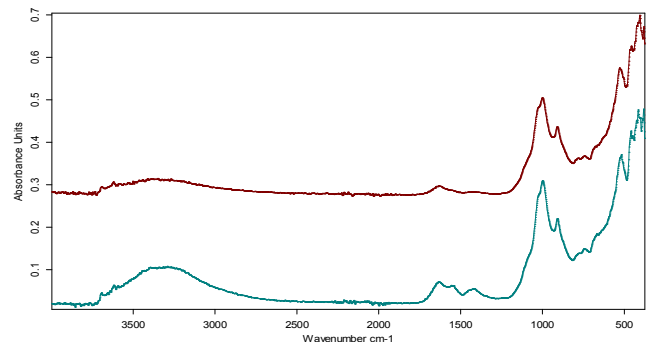


Figure 10. IR spectrum of S-TIK before and after adsorption

A comparison of the IR spectra before and after adsorption does not reveal any peak characteristic of a new bond formed. This confirms the weak interaction between the nitrate ions and the soils S-BEL and S-TIK while confirming that the adsorption mechanism is not chemisorption but rather physisorption.

Conclusion

Samples of soils of Tombel and Tiko have been used to evaluate the retention capacity of nitrate ions in aqueous solution. Adsorption process was higher in acidic medium of pH equals to 2 and was increased with increase of the initial nitrate ions concentration (20-100 mg/L). The maximum nitrate ions uptake capacity was 4.133 and 3.533 mg/g for S-TIK and S-BEL respectively. The sorbent/sorbate equilibrium was well described by the pseudo-second order kinetic and by D-K-R models for S-BEL while on S-TIK it was better describe by the pseudo-second order kinetic and Tempkin

models. Kinetics and equilibrium studies revealed that S-TIK have a good retention capacity of nitrate ions compared to S-BEL. Therefore, these samples of soil can be used to clean water contaminated by nitrates ions.

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