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Kinetics and Adsorption Isotherms of Arsenic (V) onto Natural Pozzolan

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Abstract: Adsorption of arsenic (V) from aqueous solutions onto natural pozzolan from Djoungo was studied. Experiments were carried out in batch as a function of contact time and pH at a frequency of 7 Hz. Results showed that adsorption equilibrium onto pozzolan was attained in 20 min at the pH studied and kinetics followed a pseudo-second-order model. The experimental isotherm data were analyzed using Freundlich, Langmuir and Redlich-Peterson isotherm equations. In order to determine the best-fit isotherm for each system, non-linear regressions were carried out. For this, Chi-square error was used to evaluate the data. The errors values indicated that the Redlich-Peterson isotherm was able to provide best quality fit for all the experimental data.

Key words: Arsenic (V), adsorption, pozzolan, non-linear, equilibrium

INTRODUCTION

Contamination of drinking water by arsenic has become a key environmental problem of the 21st century. Arsenic is a ubiquitous element and its presence in water due to natural and anthropogenic sources, leaching and soil erosion, leads to arsenic dissolution into the aquatic environment (Smedley and Kinniburgh, 2002). Arsenic occurs in natural waters mainly as inorganic arsenite (As (III)) and arsenate (As (V)), it does not often form in its elemental state and is more common in sulfides and sulfo salts, such as arsenopyrite, orpiment, realgar (Smedley and Kinniburgh, 2002; Meng *et al*., 2002).

Presence of high arsenic concentrations has been reported in drinking water in Taiwan, Mexico, Bangladesh, Argentina and Ghana (Litter *et al*., 2010; Ravenscroft *et al*., 2005; Mandal and Suzuki, 2002; Hossain, 2006). The maximum permissible limit of arsenic in drinking water according to USEPA and WHO guidelines is 0.01 ppm. Above this concentration, long-term drinking water causes toxic and carcinogenic effects on human beings (Saha *et al*., 1999). In other to remove arsenic from water before drinking, numerous technologies, such as coagulation, ion exchange, precipitation, membrane processes and adsorption have been adopted with varying degrees of success (Gregor, 2001; Zaspalis *et al*., 2007). Adsorption is one of the most common available technologies and it is consider as a promising technique because of its simplicity, cheaper, sludge free operation and regeneration capacity (Ramesh *et al*., 2007; Kundu and Gupta, 2006). Synthetic adsorbents developed for arsenic removal include iron oxide (Wilkie and Hering, 1996; Thirunavukkarasu *et al*., 2003; Jeong *et al*., 2007), aluminum oxide (Jeong *et al*., 2007; Lin and Wu, 2001), iron-oxide-coated polymeric material (Katsoyiannis and Zouboulis, 2002). However, other naturally abundant materials such as laterite (Maiti *et al*., 2007), zeolites (Payne and Abdel-Fattah, 2005) and pozzolan are also used (Serrano-Giomez *et al*., 2010). Arsenic (V) adsorption have been focus on a kinetics and isotherms studies using single pH and less reports has been identified on the effect of pH on the kinetics and isotherms of arsenic (V) adsorption on natural material. The aim of this study is, therefore to evaluate the efficiency of pozzolan for arsenic removal from water.

MATERIALS AND METHODS

Adsorbents: In this study, the adsorbents used are natural pozzolan from Djoungo quarry (Latitude 4°35'18"N, Longitude 9°37'48"E) Cameroon. Two types of

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pozzolan designated, hereafter as B-PZ for black pozzolan and R-PZ for red pozzolan. The rocks were grounded and the powder sieved with standardized stainless steel sieves (ISO 3310-1) and the fraction <32 μm was collected. Prior to adsorption studies, pozzolan powder was washed several times with distilled water by a sequence of mixing, settling and decanting. The washed solid fraction was then dried at 105°C for 24 h, allowed to cool in a dessicator and stored in a caped polyethylene bottles.

The chemical composition of the pozzolan used was determined by X-ray fluoresecence, XRF, Spectrometer (Axios, UK).

Nitrogen adsorption-desorption isotherms at 77 K were recorded on a step-by-step automatic home built setup. Prior to adsorption, the samples were outgassed overnight at 120°C and under a residual pressure of 0.01 Pa. Nitrogen N55 (purity 99.9995%) used for experiments was provided by Alphagaz (France). Specific Surfaces Areas (SSA) were determined from adsorption data by applying the Brunauer-Emmet-Teller (BET) equation (Brunauer *et al.*, 1938) and using 16.3 A for the cross-sectional area of nitrogen (Gregg and Sing, 1982). In the present study, the error in the determination of the SSA was estimated as ±1 $m^2 g^{-1}$. Pore size distributions were calculated on the desorption branch using the Barrett-Joyner-Halenda Method, assuming cylindrical shape pores (Barrett *et al.*, 1951).

Arsenic analysis: All the reagents used were of analytical grade. Sodium arsenate, $Na_2HAsO_4.7\ H_2O$ (Sigma-Aldrich, Germany) was used to prepare stock solutions of arsenic for establishing the calibration graph used for analysis and adsorption studies.

The determination of arsenic was carried out by a highly sensitive spectrophotometric method described by Qian-Feng and Peng-Fei (1983). This method is based on the formation of an ion-association complex between arsenoantimonomolybdenum blue and malachite green. This ion-association complex is soluble in Triton X-305, so that arsenic can be determined directly in aqueous solution at 640 nm by means of a UV-visible spectrophotometer. The UV-visible spectrophotometer used was a Jenway Model 6300 (Barloworld Scientific Ltd., UK). A calibration graph was done and gave a straight line with and R^2 value of 0.999.

Adsorption experiment: The adsorption experiments were performed in batch mode using 100 mL erlenmeyer flasks fixed to a shaker, Oscill 12 (Prolabo France). About 50 mL each of arsenic (V) solution was placed in to erlenmeyer flasks for duplicate studies and the pH of the solution in each flask was adjusted to the desired value with either 0.1 M HCl or 0.1 M NaOH solution. The ionic strength of the solutions was, also adjusted by adding KCl solution. A predetermined amount of adsorbent was then added to each flask. The flasks were fixed on the shaker and set on vibration at a frequency of 7 Hz. After a given time, the shaker was stopped and the flasks removed. The suspensions were centrifuged at 5000 g (Biofuge, Hereaus Germany) for 7 min and the supernatant was filtered on a Minisart X50 (Prolabo, France) membrane filter of porosity 0.1 μm. The amount of arsenic adsorbed (q) was calculated as the change in the aqueous-phase concentration from initial value according to Eq. 1. The blank experiment showed no detectable arsenic (V) adsorbed on the walls of the flask:

$$q = \frac{(C_0 - C)}{m} \times V \qquad (1)$$

Where:

C_0 = The initial concentration (μg L^{-1})
C = The concentration (μg L^{-1}) at contact time t (min)
V = The solution volume (mL)
m = The amount of adsorbent added (g)

RESULTS AND DISCUSSION

Adsorbent characteristics: The chemical composition of pozzolan used for arsenic removal from water in this study is presented in Table 1. The results show that the two pozzolan samples were very similar in chemical composition. It is, however observed that the amount of SiO_2, Al_2O_3 is greater in BPZ than in RPZ. But, the content of iron oxide (Fe_3O_4/Fe_2O_3) for RPZ is higher than that of BPZ. After followed in proportion MgO and CaO. The alkali (Na_2O and K_2O) constituted nearly 5%, of the total mass of oxides. This chemical composition of pozzolan was similar to that determined by other researchers (Sieliechi *et al.*, 2012; Sersale, 1980). However, these pozzolan samples were richer in aluminium oxide and iron oxide than Mexican pozzolan used by Serrano-Giomez *et al.* (2010) for arsenic adsorption. The weak loss of ignition indicates that pozzolan are very compact materials.

The nitrogen adsorption-desorption isotherms on the natural pouzzolan are shown in Fig. 1. Nitrogen adsorption on the studied pozzolan displayed similar shapes without hysteresis similar to type III isotherms in the IUPAC classification, indicating the absence of mesopores and macropores. Numerical values deduced from the adsorption isotherms showed that specific surface area of black pozzolan and red pozzolan are, respectively 2.60 and 2.68 $m^2 g^{-1}$. Pore size distributions

Table 1: Chemical composition of Black Pozzolan (BPZ) and Red Pozzolan (RPZ°, %W/W)

Composition	SiO_2	Al_2O_3	Fe_2O_3	CaO	MgO	K_2O	Na_2O	SO_3	TiO_2	Mn_2O_3	P_2O_5	P.F
BPZ	39.56	13.12	21.19	9.50	5.89	1.27	3.24	0.51	4.14	0.28	0.52	0.18
RPZ	37.63	12.90	21.79	9.87	6.45	1.57	3.59	0.51	4.48	0.56	0.51	0.15

Constituents are given in the basis of oxides

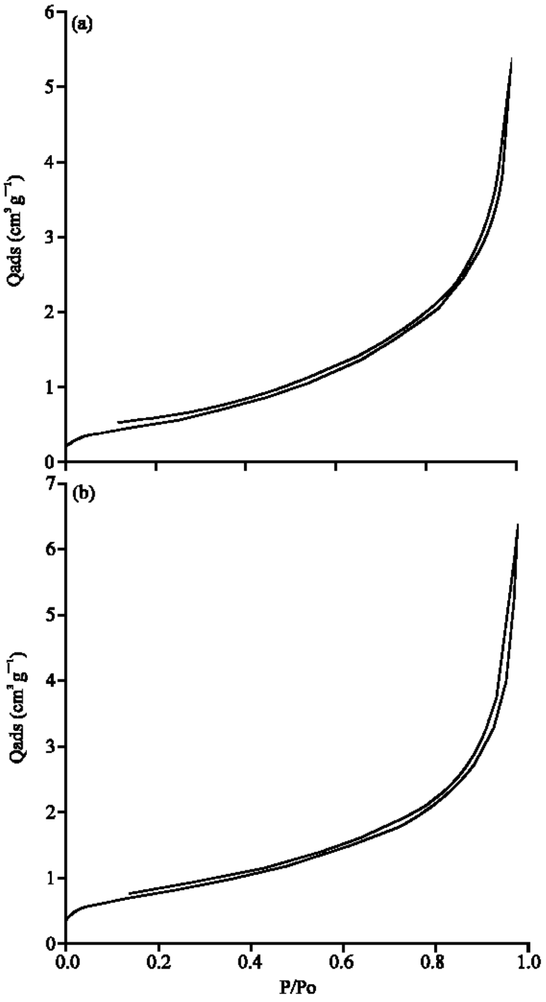

Fig. 1: Nitrogen adsorption-desorption at 77 K on: a) Black Pozzolan (BPZ); b) Red Pozzolan (RPZ)

were calculated on the desorption branch using the Barett-Joyner-Hallenda Method assuming cylindrical-shaped pores. The results indicated that pozzolan are micro porous materials with pore diameter <20 A.

Adsorption kinetics: The influence of contact time on the adsorption of arsenic onto adsorbents was investigated at constant ionic strength (10^{-3} M. KCl) and at various pH, 5, 7 and 9, respectively (Fig. 2). It is easily seen from Fig. 2 that the adsorption of arsenic on pozzolan was fast and the equilibrium was attained in <20 min at the studied pH. This implies that the diffusion rate constant of arsenic molecule is independent of the solution pH and the type of pozzolan at the studied concentration.

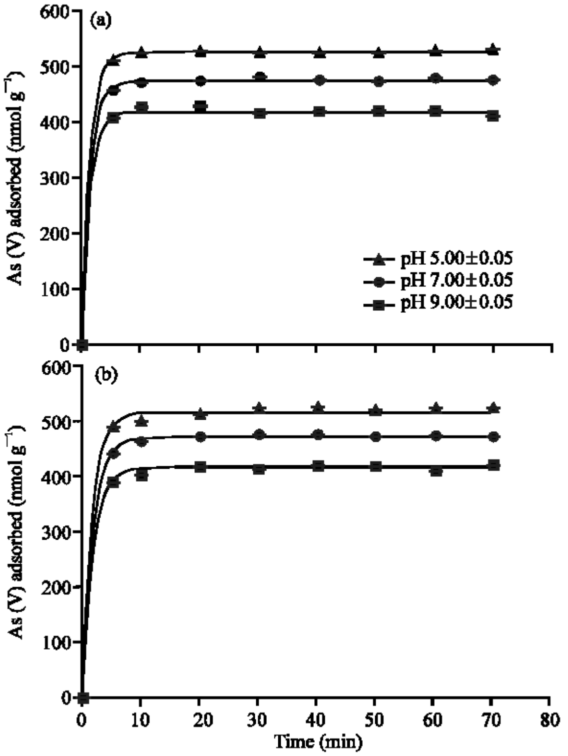

Fig. 2: Kinetics of As (V) adsorption by: a) Black pozzolan; b) Red pozzolan; pH = 7±0.05, KCl = 10^{-3} M; T = 24±2°C, initial concentrations 26.67 mmol L^{-1}

In order to investigate and to get the potential rate-controlling step, the kinetic models available in the literature have been applied to the experimental data. The model are discussed in the following sections Pseudo first-order model is given as (Jeong *et al.*, 2007; Altundogan *et al.*, 2000):

$$\log(q_e - q_t) = \log q_e - \frac{k_1}{2,303}t \tag{2}$$

Pseudo second-order kinetic model equation (Ho and McKay, 1998) is expressed as follows:

$$\frac{t}{q_t} = \frac{1}{k_2 q_e^2} + \frac{1}{q_e}t \tag{3}$$

Where:

q_e, q_t = The amount of arsenic adsorbed per unit mass ($\mu g\ g^{-1}$) of adsorbent at equilibrium and time
t = Min, respectively
K_1 = The pseudo-first rate constant ($1\ min^{-1}$)
K_2 = The pseudo-second order rate constant (g/µg.min)

Intraparticle diffusion equation (Weber and Morris, 1963) can be written as follows:

$$q = K_{int} t^{0,5} + C \tag{4}$$

Where:

K_{int} = The intraparticle diffusion rate constant ($\mu g/g.min^{1/2}$)
C = Constant

These kinetic models were used to fit experimental data. As it can be seen in Table 2, the adsorption of arsenic onto the studied pozzolan follow a Pseudo-second-order model because the experimentally measured and calculated amounts are closer, suggesting that adsorption process might be chemisorption.

In order to show the existence of intraparticle diffusion in the adsorption process, the amount of arsenic adsorbed at time t, q was plotted versus the square root of time. The adsorption of arsenic onto pozzolan is very fast making it difficult to apply this model.

Adsorption isotherms: The arsenic (V) ion adsorption was carried out at pH 5, 7 and 9. Analysis of the isotherm data is essential in order to develop an equation which accurately represents the results of batch. In addition, adsorption isotherm can be used to describe how a solute interacts with an adsorbent and is determinant in optimizing the use of adsorbent. Three isotherm equations have been applied for this study, Langmuir, Freundlich and Redlich-Peterson. The Langmuir equation is given as:

$$q_e = \frac{b q_m C_e}{1 + b C_e} \tag{5}$$

Where:

C_e = The equilibrium adsorbate concentration in solution
qmax = The amount adsorbed in a monolayer (adsorption capacity)
q_e = The amount adsorbed per unit mass of adsorbent
b = The constant binding energy

The adsorption equilibrium data was also applied to the Freundlich Model:

$$q_e = K_F \times C_e^{1/n} \tag{6}$$

Table 2: Kinetics parameters for the adsorption of arsenic (V) onto Black Pozzolan (BPZ) and Red Pozzolan (RPZ) at various pH; Kcl = 10^{-3} M, T = 24±2°C

Adsorbent	pH	$q_{e\ (exp)}$ ($\mu g\ g^{-1}$)	Pseudo-second-order		
			$q_{e\ (cal)}$ ($nmol\ g^{-1}$)	K_2 (g/nmol/min)	R^2
BPZ	5	525.2	531.20	1.88	0.999
	7	472.8	479.60	1.71	0.999
	9	416.0	430.13	1.68	0.999
RPZ	5	518.8	516.80	1.40	0.999
	7	472.0	481.33	1.31	0.998
	9	416.8	431.47	0.67	0.999

Where K_F and n are constant and considered as indicators of adsorption capacity. Freundlich parameters (K_F and n) indicate whether the nature of adsorption is favourable or unfavourable. The value of n are higher than 1 which indicates that adsorption capacity is favoured over the entire range of the studied concentration (Potgieter *et al*., 2006).

The Redlich-Peterson isotherm model was also applied. It has three parameters featuring both the Freundlich and Langmuir isotherm equations. The Redlich-Peterson (Ncibi, 2008) equation is:

$$q_e = \frac{A C_e}{1 + B C_e^g} \tag{7}$$

Where:

A and B = Redlich-Peterson constant
g = Redlich-Peterson exponent

Redlich-Peterson equation has two limiting cases which can be explained as follows. When the exponent g = 1, the Langmuir equation results, given by:

$$q_e = \frac{A C_e}{1 + B C_e} \tag{8}$$

When g = 0. Redlich-Peterson isotherm equation transforms to Henry's law equation:

$$q_e = \frac{A C_e}{1 + B} \tag{9}$$

In order to asses different isotherms and their ability to correlate with experimental results, the theoritical plots from each isotherm have been fitted with the experimental data for adsorption of arsenic on pozzolan. Figure 3 shows Langmuir and Redlich-Peterson isotherm with experimental data at the various pH.

The effect of isotherms shapes were investigated in order to predict whether an adsorption system is favorable or unfavorable. The essential quality of the Langmuir isotherm can be expressed by means of R_L, a

Table 3: Adsorption isotherm parameters for Freundlich, Langmuir and Redlich-Peterson Models

		Black pozzolan			Red pouzzolan		
Isotherms	Cte	pH 5	pH 7	pH 9	pH 5	pH 7	pH 9
Freundlich	K (nmol g^{-1})	85.730	64.130	58.130	93.600	78.930	47.600
	n	3.640	3.380	3.270	3.800	3.660	3.020
	R^2	0.955	0.961	0.964	0.951	0.952	0.975
	χ^2	3.270	3.210	2.690	4.000	3.730	1.850
	Qmax (nmol g^{-1})	557.600	531.330	438.930	560.930	506.400	461.070
Langmuir	b (L $nmol^{-1}$)	0.200	0.160	0.187	0.227	0.213	0.147
	R_L	0.030	0.040	0.030	0.030	0.030	0.040
	R^2	0.999	0.994	0.984	0.999	0.999	0.994
	χ^2	0.040	0.490	0.980	0.080	0.030	1.060
Redlich-Peterson	g	0.999	0.994	0.923	0.999	0.999	0.913
	B (L $nmol^{-1})^g$	0.213	0.173	0.400	0.227	0.213	0.360
	A (L g^{-1})	0.654	0.495	0.587	0.707	0.621	0.509
	R^2	0.999	0.994	0.989	0.999	0.999	0.995
	χ^2	0.040	0.460	0.620	0.090	0.030	0.450

Experimental conditions studied

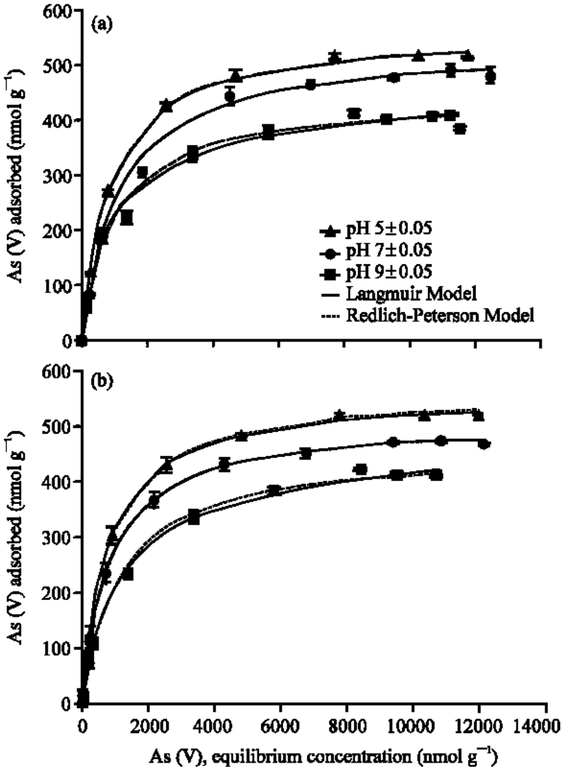

Fig. 3: Isotherm data and fitted model equation for adsorption of As (V) on: a) Black pozzolan; b) Red pozzolan; KCl = 10-3 M; T = 24±2°C

dimensionless constant refferred as a separation factor or equlibrium parameter. R_L was calculated by using the following equation:

$$R_L = \frac{1}{1 + bC_0} \tag{10}$$

If R_L values lie between 0 and 1, adsorption process is considered to be favorable (Mahramanlioglu *et al.*, 2002; Hall *et al.*, 1966).

The Chi-square test was also use to estimated the best fitting model. It is the sum of the squares of the differences between the experimental data and theoritically predicted data from models:

$$\chi^2 = \frac{\left(q_{exp} - q_{cal}\right)^2}{q_{cal}} \tag{11}$$

The Langmuir, Freundlich and Redlich-Peterson parameters for the adsorption of arsenic (V) onto pozzolan are listed in Table 3. Those parameters were determined by non-linear method using Tablecurve 2D version 5.01. It is evident from Table 3 and Fig. 3 that the adsorption of arsenic onto pozzolan fitted the Redlich-Peterson Model as indicated by the by high coefficient of correlation ($R^2 \approx 1$) and lower value of Chi-square (χ^2). It can then concluded that adorption of arsenic (V) on pozzolan progresses from monolayer to multilayer adsorption on heteregeneous surface (Abdullah *et al.*, 2009). Morever, the separtion factor (R_L) calculated for all adsorbent varied between 0 and 1, confirmig favorable adsorption under the experimental conditions studied (Table 3).

CONCLUSION

In the present study, removal of arsenic (V) from aqueous solution was achieved using pozzolan as an adsorbent. Kinetic studies show that equilibrium time is attained in 20 min. Experimental data can be described adequately by a pseudo-second order kinetic model. Redlich-Peterson isotherm model was found to fit the equilibrium data well.

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