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Arsenic adsorption on natural minerals

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Abstract

Natural minerals are used to remove Arsenic (As) from aqueous solution in this study. The influence of suspension pH, temperature, agitation speed and arsenic dosage on arsenic removal is investigated in a series of batch adsorption experiments. In addition, empirical relationships are developed between % As (V) removal and adsorbent, adsorbate dose. Thermodynamic parameters are evaluated from temperature experiments. The kinetic is found to be pseudo second order in nature. The negative value of free energy change indicates the spontaneity of adsorption process. The adsorption of As (V) on natural minerals is found to be pore diffusion controlled in addition to surface adsorption.

Keywords: Adsorption, Arsenic, Bauxite, Iron ore, Manganese ore, Nickel laterite.

Introduction

The occurrence of arsenic (As) in the raw water supply especially in ground water is a significant problem in Bangladesh and other countries like Argentina, Chile, India and Mexico. Although As concentration in most of the ground water is less than 10 µg l^{-1}, the concentration like 5 mg l^{-1} is also observed in some of the affected areas [1]. Since the international standard for drinking water recommended by World Health Organization (WHO) is 10 µg l^{-1}, the removal of excess As should be carried out by following some suitable methods. Adsorption of As on the surface of oxides/hydroxides can offer such. Several naturally occurring oxides/hydroxides of metals are often used as the low cost material for arsenic mitigation. The surfaces of the oxides/hydroxides are positively charged in natural systems due to protonation of surface sites [2]. These play an important role in the adsorption of anions, which tend to adsorb on positively charged surfaces and the adsorption mainly occurs at acidic/neutral pH.

The whole range of iron minerals starting from amorphous ferrihydrite to hematite, are capable of adsorbing As (III) and As (V), as arsenite and arsenate. The arsenite and arsenate adsorption kinetics on ferrihydrite ($FeOOH, 5H_2O$) is reported [3]. Besides that, As (V) is also reported to be adsorbed by iron coated sand as a medium in small system or home treatment units [4,5].

A low cost ferruginose manganese ore (FMO) has been studied for the removal of As (V) from ground water. Once adsorbed, As (V) does not get desorbed even on varying the pH in the range of 2-8. Traces of As (V) can be selectively removed from the drinking water by natural manganese dioxides in column experiments [6,7].

Removal kinetics of both the forms of As from aqueous media on modified alumina has been studied by Balaji et al. by using modified alumina, so as to attain maximum contamination level (MCL) at faster rate [8]. A process has been developed to remove both As (III) and As (V) from contaminated water and waste water by using activated alumina as the adsorbent [9].

Natural minerals are the largest source of oxy-hydroxides. These minerals/ores currently have limited industrial use. These are cheap (<100 INR/t) and are abundantly available. There is limited information available on their capability to adsorb As and in addition, their ability to adsorb As is dependent on their geographic location and mineralogy. As is most effectively removed or stabilized when it is present in pentavalent arsenate form i.e., As (V) [10]. Penta valent species predominates and are stable in O_2 rich aerobic environment, whereas, arsenite i.e., As (III) predominates in moderately reducing anaerobic environment such as ground water [11]. Therefore, in this work four major sources of minerals of eastern India, namely nickel laterite, bauxite, iron ore slime and lean manganese ore are selected as adsorbents for detailed As (V) adsorption studies in the laboratory scale using synthetic As (V) solution.

2. Materials and methods

2.1. Adsorbate

Standard 1000 mg l^{-1} As(V) solution (as As_2O_5) supplied by E. Merck, Germany is diluted to desired level and all the experiments are carried out using the same.

2.2. Adsorbent

For the present study, the following adsorbents found in the Odisha State of India are used.
(i) Nickel laterite is collected from Sukinda area
(ii) Iron ore slime from Keonjhar district,
(iii) Manganese ore from Roida Sector, Barbil,
(iv) Bauxite ore from Panchapat Mali,
All these materials are powdered and dried at 110°C overnight. The 150 µm size fractions of all the materials are taken for adsorption studies.

2.3. Adsorption studies

All the adsorption studies are carried out in 250 ml glass conical flasks. The solution volume used in each experiment is 50 ml. Stirring is done by a horizontal Julabo Shaker (Julabo-SW-20C). The leaching of any type of pollutants during shaking has been neglected here, as the adsorption studies are being carried out in neutral medium in most of the cases. The pH is measured using combined glass electrode. The pH adjustment is made by using either dilute sodium hydroxide or dilutes nitric acid as necessary. After the required contact time (180 minutes for bauxite and 30 minutes for other minerals), the contents are filtered using Whatman 42 filter paper. It is observed that no As (V) adsorption is there on the filter paper itself.

The experimental loading (mg g^{-1}) = $(C_o-C_e)V/W$

Where, C_o and C_e are the initial and equilibrium concentrations (mg l^{-1}) of As (V) in solution, V is the volume (l) and W is the weight (g) of the adsorbent.

2.4. Analyses

The arsenic concentration in aqueous solution is determined by UV-VIS Spectrophotometer at 840 nm wavelength using molybdate method [12]. For this method, As (V) species are first reduced to As (III) with ascorbic acid followed by complexation with ammonium heptamolybdate. The limit of determination of this method is 0.05 mg l^{-1}.

3. Results and discussion

3.1. Characterization of the minerals

According to X-Ray Diffraction study, bauxite consists of Gibbsite, Muscovite, Kaolinite, Quartz and Siliminite. Ni laterite consists of Goethite, Hematite and quartz. Iron ore slime consists of Hematite, Goethite, Iron silicate, Quartz and Nordstrandite, where as manganese ore consists of Nontronite, Jacobsite and Quartz. It indicates that these minerals contain either oxides or hydroxides of metals. Other physical parameters like point of zero charge (pzc), surface area and density are shown in Table 1.

Table 1: Physical and Chemical Characteristics of the minerals studied

Constituents	**Weight(%)**			
	Bauxite	Ni laterite	Fe ore slime	Mn ore
SiO_2	13.8	36.5	3.32	4.23
Al_2O_3	54.87	5.6	3.7	6.32
Fe_2O_3(Total Fe)	6.98	39.4	89.21	26.25
FeO	--	5.0	--	1.59
Cr_2O_3	0.08	4.4	--	0.32
MgO	0.02	1.6	--	0.6
MnO	0.037	0.23	--	53.32
CoO	--	0.03	--	--
TiO_2	2.0	--	--	--
P_2O_5	0.12	--	--	--
CaO	0.009	0.41	--	--
Loss on ignition, %	22	6.23	3.77	6.5
Surface area, m^2 g^{-1}	31.57	25.6	13.2	21.4
Density, g l^{-1}	2.5	2.5	3.33	2.85
pzc	7.2	6.8	7.0	6.8

3.2. Arsenic adsorption studies

3.2.1. Effect of contact time

Time of contact between adsorbate and adsorbent plays an important role in the adsorption process. The time needed to attain equilibrium is important to predict the efficacy and feasibility of an adsorbent for its use in pollution control. The effect of contact time on As (V) adsorption is investigated by running adsorption experiments for four hours. The results are shown in Figure 1. The experimental conditions are mentioned in the figure legend. During the removal of As (V) by adsorption on different naturally occurring materials studied, it has been found that the uptake is increased up to 180 and 30 minutes for bauxite and other adsorbents respectively and thereafter it attains a stationary state. So, 180 minutes and 30 minutes are considered to be the optimum contact time for bauxite and other adsorbents respectively. It has been shown from Figure 1 that % of As (V) adsorption on different minerals follow the following trend.

Bauxite > Nickel laterite > Iron ore slime > Manganese ore

Since the surface area of bauxite is the highest as shown in Table 1, it may be the cause of its high adsorption capacity in comparison to the other minerals studied.

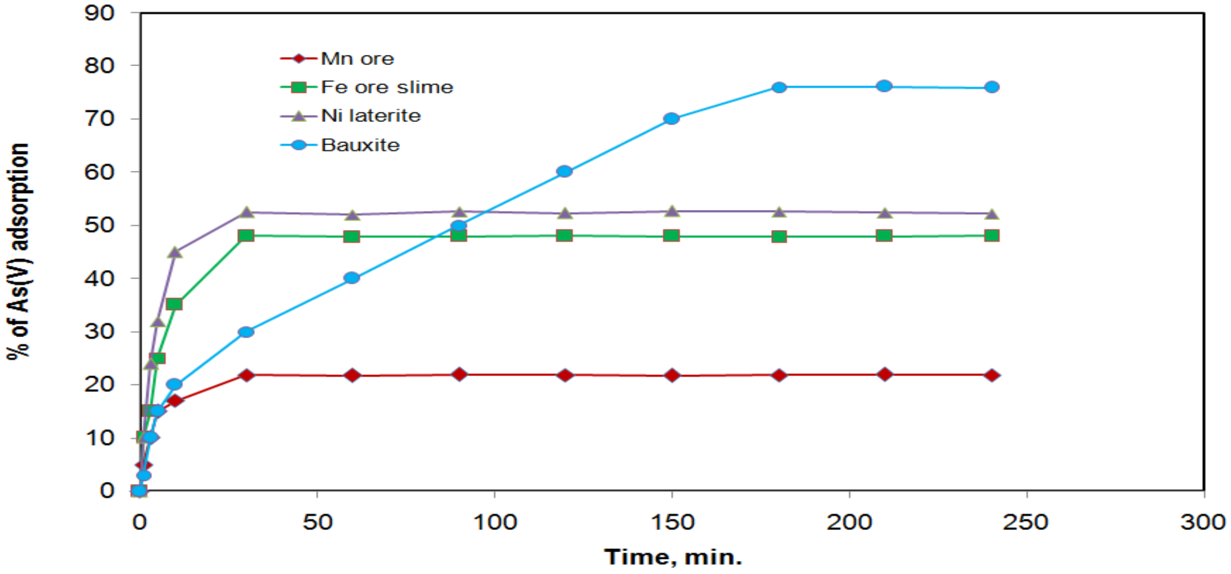

Figure 1: Effect of contact time on the adsorption of As (V) [Conditions : As(V) : 10 mg l^{-1}, pH : 7.0, Agitation speed : 150 rpm, Slurry density (optimum) : 2%]

3.2.2. Effect of pH

The initial pH of the solution is varied from 6.0 to 8.0. This pH range is chosen because it is the typical ground water pH in India/Bangladesh, where ground water is generally As contaminated. The results are shown in Figure 2. It is observed from the figure that As (V) adsorption increases from pH 6.0 to 7.0 and after pH 7.0, there is no further increase in the adsorption percentage. The adsorption on bauxite surface is almost pH independent. For all the minerals studied, pH 7.0 is the optimum pH of adsorption. Therefore, it is a great advantage that these adsorbents can be used at a neutral/natural pH without hampering its adsorption capacity.

The effect of pH on adsorption process depends on several factors such as nature of the adsorbent surface, speciation of As (V) species and the acid/base used for the adjustment of pH of the solution. In order to explain the pH effect, it is necessary to examine the various mechanisms such as electrostatic attraction/repulsion, chemical interaction and ion exchange, which are usually responsible for adsorption of a species on any adsorbent surface. Around pH 7.0, As (V) is generally present either in the form of $H_2AsO_4^-$ (below pH 7.0) or, $HAsO_4^{2-}$ (above pH 7.0). The pzc value of different naturally occurring materials are nearly 7.0 (shown in Table 1) indicates that the surface is positively charged at low pH i.e., below pzc and should be quite favourable for the uptake of negatively charged arsenate species. Particularly, oxides, oxy hydroxides and silicate mineral surfaces in contact with water are electrically charged because of ionization of MOH functional group (M represents a metal ion at the solid surface). The magnitude and sign of this surface charge varies with the solution pH [2]. So, at low pH, the interaction between positively charged metal surface and negatively charged arsenate species can be represented by the following equation.

$$MOH + L^- + H^+ \leftrightarrow M\text{-}L + H_2O$$

Where, L^- is the negatively charged arsenate species and ML is the adsorbed species.

But, according to our observation, this is not the case and As (V) adsorption at lower pH is comparatively less. Therefore, adsorption of As (V) species on naturally occurring surface under natural condition cannot be explained on the basis of electrostatic attraction alone. Specific chemical interaction and the surface complexation also have an important role in the adsorption of As (V) on naturally occurring materials.

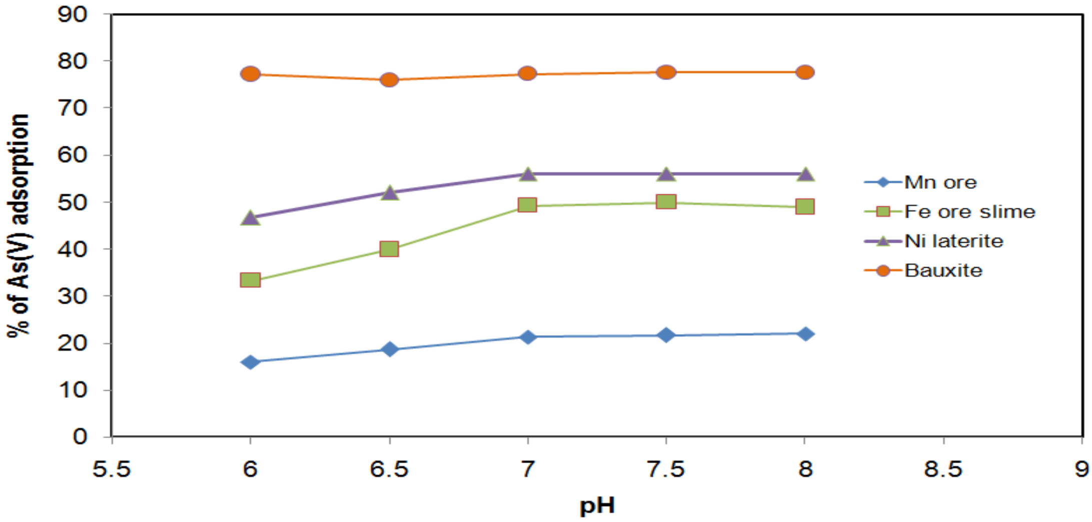

Figure 2: Effect of pH on the adsorption of As (V) [Conditions : As(V) : 10 mg l^{-1}, Time: 180 minutes for bauxite and 30 minutes for others, Agitation speed : 150 rpm, Slurry density (optimum) : 2%]

3.2.3. Effect of initial As (V) concentration

The effect of initial As (V) concentration (1 to 10 mg l^{-1}) on the As (V) removal efficiency has been studied. With the increase of initial As (V) concentration at a fixed adsorbent amount in the solution, the % of As (V) removal for different naturally occurring materials decreases, because the number of active adsorption sites are limited to adsorb the available adsorbate ions whereas, loading, mg g^{-1} shows a reverse trend (Figure not shown). The following empirical models are developed correlating the % of removal of As (V) and initial As (V) concentration, in mg l^{-1} [13]. It gives a clear idea about the removal (%) at all initial As (V) concentrations.

Removal (%) = KC_o^n (1)

Where, C_o = Initial As (V) concentration, mg l^{-1}

K = Constant

Figure 3 shows a plot between ln (Removal, %) versus ln C_o. The K and n values are obtained from the intercepts and slopes respectively. Based on K and n values, the following empirical equations can be developed which would indicate the extent of adsorption based on initial As (V) concentration at an adsorbent concentration of 20 g l^{-1}.

Bauxite : As(V) removal, % = $89.12\ C_o^{-0.25}$

Ni laterite : As(V) removal, % = $89.12\ C_o^{-0.26}$

Fe ore slime : As(V) removal, % = $60.9\ C_o^{-0.28}$

Mn ore : As(V) removal, % = $51.42\ C_o^{-0.82}$

In the process of adsorption, after specific time, a dynamic equilibrium is achieved between the adsorbate present in the solid (on the surface of adsorbent) as well as in the aqueous phase. The distribution of adsorbate between the two phases can be expressed in terms of the adsorption isotherm. The adsorption isotherms can be explained through Freundlich or Langmuir isotherms represented by equations 2 and 3 respectively.

$\ln(X/M) = \ln K_1 + 1/n \ln C_e$ (2)

$X/M = b\ C_e / (1/a + C_e)$ (3)

Where, X/M = Amount of As (V) adsorbed per unit mass of sorbent (mg of sorbate / g of sorbent)

C_e = Equilibrium concentration of As(V) in the solution, mg l^{-1}

K_1 = Constant which is a measure of adsorption capacity
1/n = Measure of adsorption density
a = Constant related to the energy of adsorption and
b = Monolayer adsorption capacity

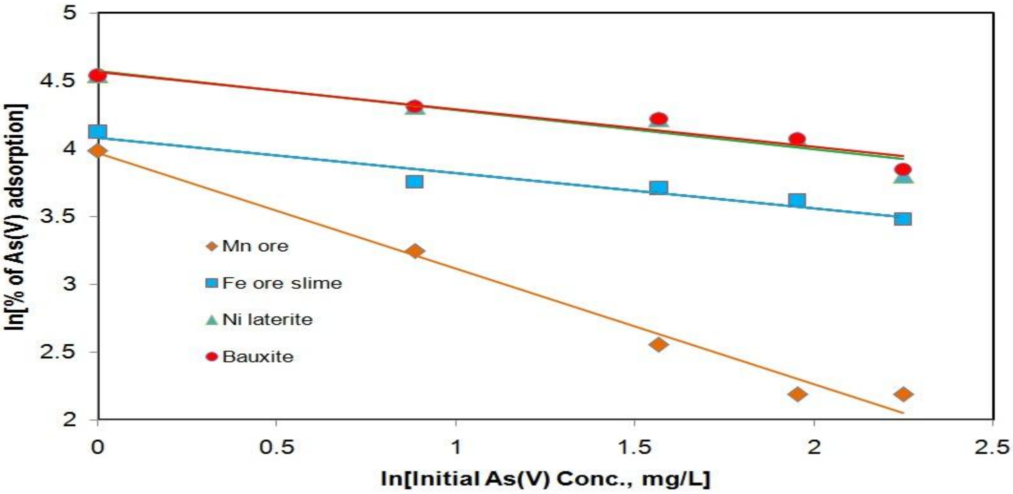

Figure 3: Determination of dependence factors for As (V) concentration variation

A graph of ln X/M versus lnC_e gives a straight line in case of Freundlich sorption isotherm, where slope and intercept are 1/n and ln K_1 respectively. Similarly, for Langmuir isotherm, a straight line would be obtained when C_e/X/M is plotted against C_e. Langmuir's constants (a and b) can be calculated from the slope (=1/b) and intercept(=1/ab) of the same plot. The coefficient of determination values (R^2) and the isotherm constants for both the isotherms are given in Table 2. It shows that the Langmuir plot is more appropriate for these systems. Monolayer adsorption capacity, b, for bauxite, nickel laterite, iron ore slime and manganese ore are found to be 0.51, 0.55, 0.40 and 0.09 mg g^{-1} respectively. These values are comparable with the adsorption capacity for iron oxide (0.65 mg g^{-1}) and other low cost materials like waste iron slag (0.0476 mg g^{-1}) and waste filter sand (0.25 mg g^{-1}) reported earlier [14,15]. Monolayer adsorption capacity of the system shows that manganese ore is the less effective sorbent, whereas, bauxite and nickel laterite are the best.

Table 2: Freundlich and Langmuir's constants along with their correlation coefficients for the minerals studied

Minerals	**K**	**n**	**R^2**	**a**	**b**	**R^2**
Nickel laterite	0.22	2.1	0.98	1.03	0.51	0.99
Bauxite	0.23	2.2	0.98	0.88	0.55	0.97
Fe ore slime	0.10	1.9	0.95	0.33	0.40	0.81
Manganese ore	0.06	7.1	0.67	1.12	0.09	0.92

The separation factor or equilibrium parameter, R_L, can be predicted from Langmuir's plot [16].

$$R_L = 1/\ (1+aC_o) \qquad (4)$$

Where, a = Langmuir's constant
C_o = Initial As (V) concentration, mg l^{-1}
and R_L = Dimensionless constant

R_L values for the entire adsorbents lie within 0-1 for all the concentration ranges studied indicating that the adsorption is favorable [17].

3.2.4. Effect of mineral concentration

With the increase of adsorbent concentration, the percentage of As (V) adsorption should increase proportionally, but experimentally it has been found that % of As (V) adsorption increase with the adsorbent concentration up to a certain slurry density and there after no change is observed with the addition of extra adsorbent. Because it seems that at a particular slurry density, equilibrium occurs between adsorbate and adsorbent and once equilibrium is established, the adsorbate present in the aqueous phase does not respond to the extra added adsorbent.

Here also empirical relationships can be established by plotting a graph between ln (removal %) and ln (mineral concentration, g l^{-1}) for each of the minerals studied. The K and n values of equation 1 are obtained from the intercepts and slopes respectively and the equations are
As(V) removal, % = 10.4 (Bauxite, g $l^{-1})^{0.5}$
As(V) removal, % = 12.73 (Ni laterite, g $l^{-1})^{0.47}$
As(V) removal, % = 13.54 (Fe ore slime, g $l^{-1})^{0.34}$
As(V) removal, % = 2.49 (Mn ore, g $l^{-1})^{0.6}$

By using these equations, loadings, mg g^{-1} at different mineral concentrations are calculated and the calculated loadings are found to be almost equal to the experimental loadings. Figure 4 shows the effect of mineral concentration on loading for different minerals studied.

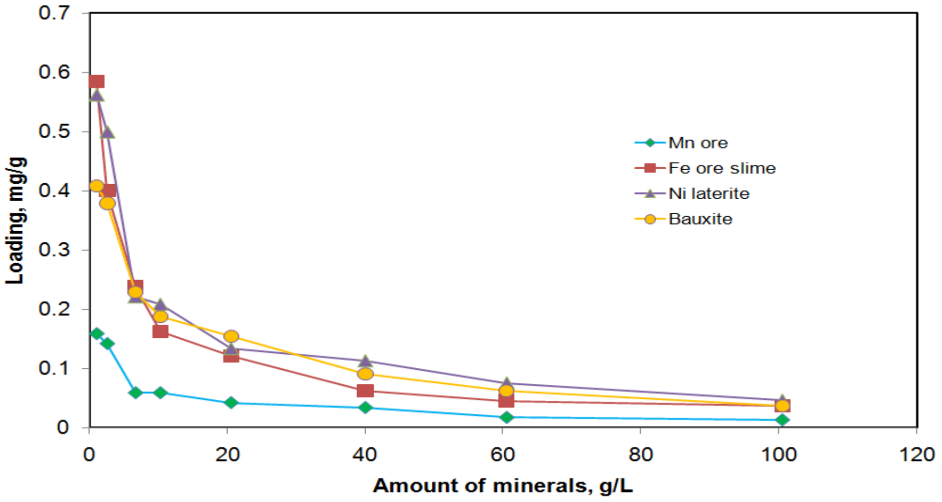

Figure 4: Effect of mineral concentration on loading [Conditions: Time: 180 minutes for bauxite and 30 minutes for the others, As (V) : 10 mg l^{-1}, pH : 7.0, Agitation speed : 150 rpm]

3.3. Thermodynamics of adsorption

The temperature of the system is varied from 30-60° C. It is observed (Figure 5) that for manganese ore, iron ore slime and bauxite, the percentage of sorption increases with the increase of temperature up to 50°C and then it decreases at higher temperature. However, nickel laterite shows very little variation in arsenic adsorption with respect to temperature. All the four samples show maximum adsorption at 50°C.
The data are analyzed according to the Arrhenius equation,

$$k = Ae^{-E/RT} \qquad (5)$$

Where, k = Reaction rate constant
A = Frequency factor
E = Activation energy
T = Temperature, K
R = Universal gas constant

The activation energy is determined from the Arrhenius plot (a plot between ln k and 1/T) for all the four naturally occurring minerals and is shown in Table 3. The low activation energy in the range (13-67) kJ $mole^{-1}$ indicates the ease of sorption reaction. These values are comparable to the activation energy obtained during Zn (II) adsorption using activated carbon i.e., 25.7 kJ $mole^{-1}$ [18].
The values of ΔH^o and ΔS^o are calculated from the slopes and intercepts of the plots of ln K_D versus 1/T [19] by using the relation.

$$\ln K_D = \Delta S^o/R - \Delta H^o/RT \qquad (6)$$

where, K_D = Distribution coefficient
T = Temperature, K
R = Universal gas constant

The free energy of specific adsorption ΔG^o is calculated from the relation

$$\Delta G^o = \Delta H^o - T\Delta S^o \qquad (7)$$

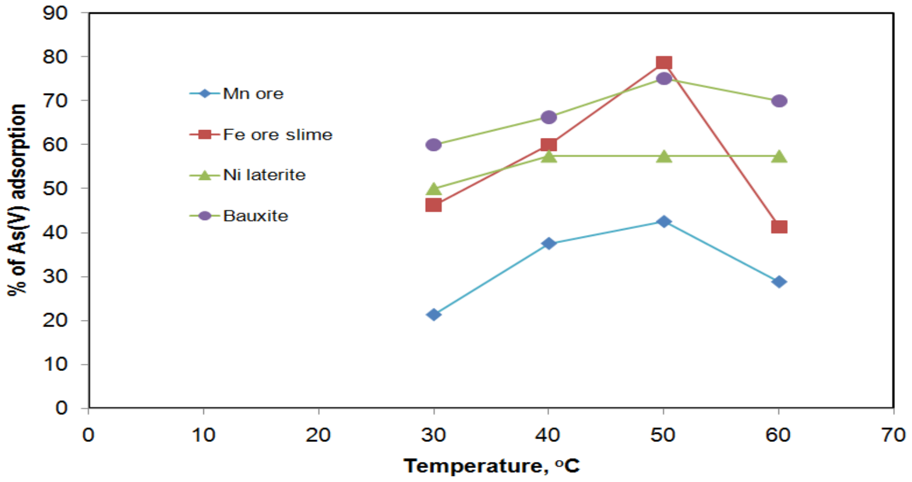

Figure 5: Effect of temperature on percentage of As (V) adsorption [Conditions: As (V) concentration: 10 mg l^{-1}, pH: 7.0, Slurry density (w/v): 2%, Time: 180 minutes for bauxite and 30 minutes for the others, agitation speeds: 150 rpm]

The calculated value in Table 3 shows that the activation energy (ΔE^o) is almost equal to the total heat content of the system (ΔH^o). So, the reaction is feasible and no extra energy is required for the commencement of the reaction. The positive values of the enthalpy change suggest the reaction to be endothermic in nature. Experimentally, it has also been found that with increasing the temperature of the system, the extent of adsorption increases. The negative value of the free energy change ΔG^o indicates the spontaneity of the adsorption process.

Table 3: Thermodynamic parameters for As(V) adsorption on different minerals

Minerals	**Temp., °C**	**ΔE^o, kJ mole^{-1}**	**ΔH^o, kJ mole^{-1}**	**ΔS^o, kJ mole^{-1}**	**ΔG^o, kJ mole^{-1}**
Fe ore slime	30	67.53	69.52	0.23	-0.17
	40				-2.47
	50				-4.77
	60				-7.07
Mn ore	30	40.24	41.24	0.13	1.85
	40				0.55
	50				-0.75
	60				-2.05
Ni laterite	30	13.58	13.90	0.05	-0.34
	40				-0.81
	50				-1.28
	60				-1.75
Bauxite	30	33.3	34.28	0.12	-2.07
	40				-3.27
	50				-4.47
	60				-5.67

3.4. Adsorption kinetics

3.4.1. Determination of the order of the reaction

The first order Lagergren's equation is used to determine the rate of the reaction [20]. The equation is

$$\log (q_e-q_t) = \log q_e - K_{ad}.t/2.303 \qquad (8)$$

where, K_{ad} = Rate constant of adsorption, q_e = Loading, mg g^{-1} at equilibrium, q_t = Loading, mg g^{-1} at any time t and t = Time, min

When log (q_e-q_t) is plotted against t, K_{ad}, the rate constant of adsorption would be obtained from the slope of the straight line. The result is shown in Figure 6. A pseudo 2nd order equation has been suggested as being more appropriate for describing this type of adsorption [21]:

$$1/ q_t = 1/2kq_e^2 + t/q_e \qquad (9)$$

The initial rate of adsorption (R) = $2kq_e^2$ is obtained from the intercept of the graph plotted between 1/ q_t versus t (Figure not shown). Table 4 shows initial sorption rates for the four materials studied.

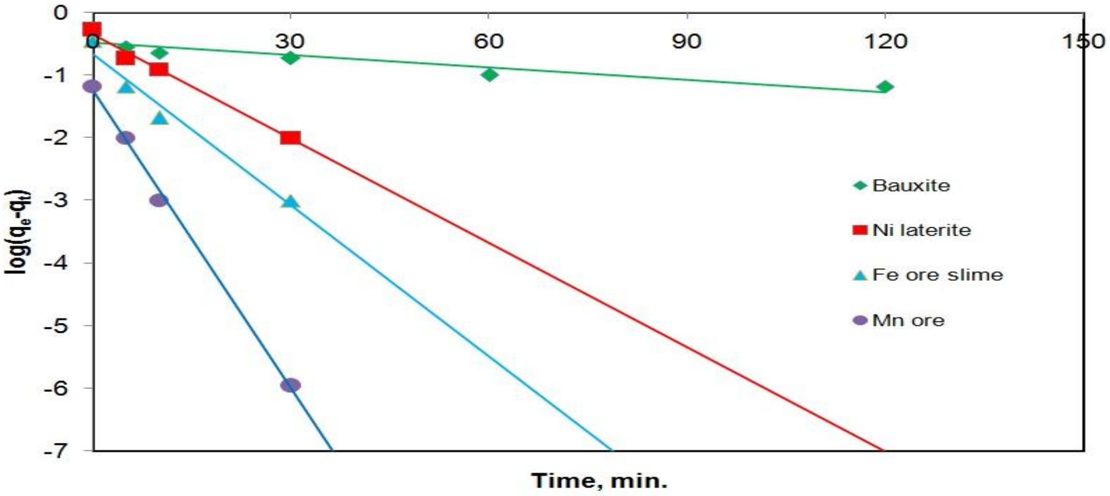

Figure 6: Lagergren's plot

Table 4: Initial sorption rates of As(V) adsorption on different minerals studied

Minerals	**Initial rates, (mg g^{-1} min^{-1})**
Bauxite	0.22
Ni laterite	0.24
Fe ore slime	0.16
Mn ore	0.066

3.4.2. Intraparticle diffusion study

When the adsorbent is porous, pore diffusion is also expected in addition to surface adsorption. The rate constants of intra particle transport (K_{id}) can be calculated from the Weber Morris equation [22]. The equation is

$$Q = K_{id}\, t^{0.5} \qquad (10)$$

Where, Q = Amount of As (V) adsorbed in mg g^{-1}

t = Time in minute

Accordingly, the graphs are plotted between Q and $t^{0.5}$ and are shown as Figure 7 and K_{id}, the rate constants for intraparticle diffusion are determined from the slopes of the linear portion of the respective plots and are shown in Table 5. The linearity of the graph indicates the reaction to be pore diffusion controlled in nature.

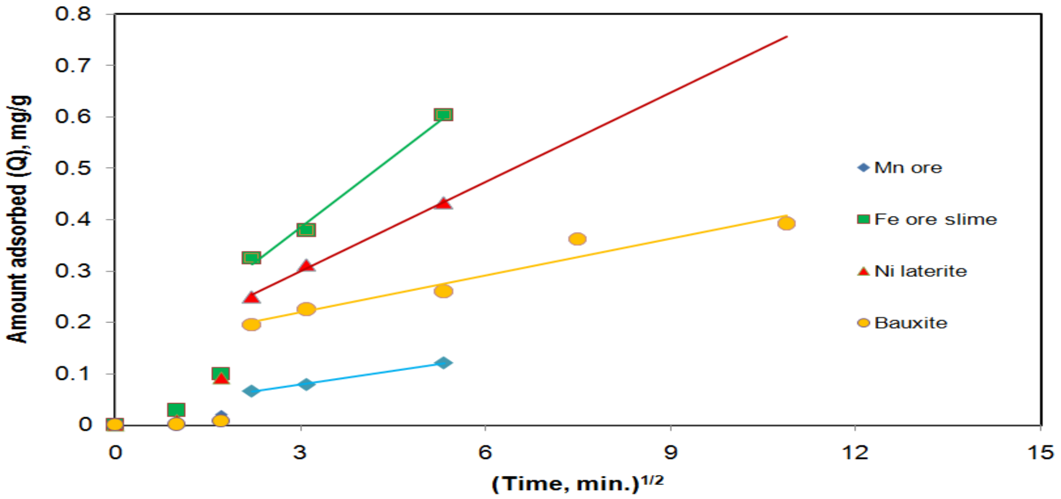

Figure 7: Determination of rate constants for pore diffusion

3.4.3. *Mass transfer analyses*

Usually, the process of adsorption on any adsorbent surface occurs following a three step model.

i. Mass transfer of solute from the aqueous phase onto the solid surface
ii. Adsorption of solute onto sites
iii. Intra particle diffusion of solute via either a pore diffusion model or a homogeneous solid diffusion model.

The first step has been assumed to be the slowest step with respect to rest of the steps, so it is the rate determining step in any kinetic study. The mass transfer equation [17] can be shown as

$$\ln [C_t/C_o-1/(1+mK_L)] = \ln[mK_L/(1+mK_L)] - [(1+mK_L)/mK_L]\, \beta S_S\, t \qquad (11)$$

where, m= mass of the adsorbent (w/v) in g l^{-1}

S_S = Outer surface of the adsorbent particle per unit volume per particle free slurry, (cm^{-1}) = $6m/d_P P_P(1-e_P)$
K_L = Langmuir constant (l g^{-1})
β = Mass transfer coefficient, cm s^{-1}
d_P = Diameter of the adsorbent particle (cm)
P_P = Density of the adsorbent (g l^{-1})
e_P = Porosity of the adsorbent particle

The value of the mass transfer coefficients (β) is determined from the slopes and intercepts of the straight line plots, ln [C_t/C_o-1/ (1+mK_L)] versus t as shown in Figure 8. The linearity of the graph indicates the process to be diffusion controlled in nature. Diffusion rate (βS_S) having the dimensions of 1st order rate constant (min^{-1}) suggests the process to be diffusion controlled in nature. Assuming S_S to be BET (Brunauer, Emmett and Teller theory for multilayer adsorption) surface area, the values of mass transfer coefficients (β) are calculated and are shown in Table 5.

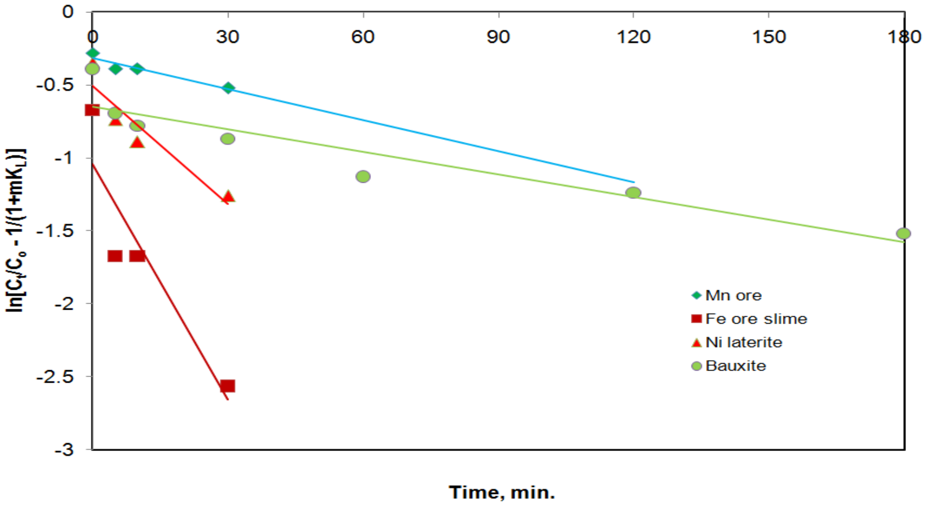

Figure 8: Determination of mass transfer coefficients (β)

Table 5: Adsorption, Intra particle transport and Diffusion controlled rate constants along-with mass transfer coefficients for different minerals studied

Minerals	**K_{ad}, min^{-1}**	**K_{id},mg g^{-1} min^{-1}**	**βS_S, min^{-1}**	**β, cm min^{-1} * 10^8**
Bauxite	0.014	0.0236	0.003	0.33
Ni laterite	0.124	0.0562	0.016	2.5
Fe ore slime	0.189	0.0866	0.044	9.97
Mn ore	0.373	0.0158	0.008	1.36

Conclusion

As (V) adsorption on natural minerals follows a trend bauxite > nickel laterite > iron ore slime > manganese ore. 30 minutes contact time is optimum for nickel laterite, iron ore slime and manganese ore, whereas, 180 minutes contact time is best for bauxite. An empirical model has been developed and thereafter % of adsorption is calculated. A good correlation is observed between theoretical and experimental % of adsorption. The positive and negative values of the enthalpy and free energy changes suggest the adsorption reaction to be endothermic and spontaneous in nature respectively. These materials are comparable to the other adsorbents used for metal removal as it is evidenced from their activation energies. Although As can be effectively removed by this method, the disposal of As enriched minerals is a serious problem. The methods frequently used for other metals may not be suitable here, as arsenic oxides are volatile and the process is not cost effective due to limited market of As. So, the attractive option for treating As wastes is encapsulation through solidification/stabilization followed by disposal in secure landfills [10]. If the As enriched minerals would be disposed safely, then the removal of As by using oxide/hydroxide minerals would be an eco friendly and cost effective method.

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